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APPLICATION OF OIL AGGLOMERATION FOR EFFLUENT CONTROL FROM COAL CLEANING PLANTS

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Agglomeration	Oil Agglomeration	14G
Coal Preparation	Coal Cleaning	08I
Sodium Hydrosulfide	Black Waters	07B

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ABSTRACT

This study demonstrates the potential applicability of the oil agglomeration technique for the control of black water effluents from coal cleaning plants processing four different coals. Removal and recovery of the coal from each of the black waters produced aqueous suspensions of mineral matter that settled more rapidly than the original black water. The sediment recovered from agglomeration appears to be less prone to acid generation during aeration than the total black water sediment.

The ash and sufur content of the coal recovered by agglomeration is less than that of the cleaned coal. The quality of the recovered coal can be improved by chemical treatment of the sediment before agglomeration. Sodium sulfide appears to be one of the better agents to use because of the simplicity of the treatment process. Such pretreatment of the sediment can reduce the pyrites by up to 50 percent in the recovered coal over that without pretreatment. even greater reductions in pyrite and ash are realized after pretreatment when the amount of oil used for agglomeration is reduced from 10 to about 2 percent and a two-stage air-float separation is used to recover the agglomerated coal.

The cost of the oil-agglomeration recovery of fine coal from coal preparation effluent streams would approximate $18 to $22 per ton of coal recovered, assuming an oil price of $.90 per gallon.

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SECTION 1

INTRODUCTION

Physical coal cleaning (PCC) processes have been used worldwide to upgrade coal quality, usually by elaborate systems to produce sulfur-free metallurgical coals and more simple systems to remove ash-forming minerals from coals used for boiler fuels. In the mid-1960's the U.S. Environmental Protection Agency began programs to determine the usefulness of PCC for reducing sulfur oxide emissions from electric utility boilers. In one of these programs the U.S. EPA worked with the U.S. Bureau of Mines (BOM) in finding answers to three basic questions: (1) Which U.S. coals used for steam generation are amenable to desulfurization by PCC techniques? (2) How effectively does commercially available equipment remove sulfur? (3) How efficient are processes that could recover coal values normally rejected with the mineral matter during coal cleaning and thereby increase the degree to which coal could be economically cleaned using physical methods?

The status of physical coal cleaning technology covered by the EPA-BOM activities and the work of others were reviewed recently by Battelle's Columbus Laboratories (EPA Contract No. 68-02-2163) [1]. The report just referenced deals with one of a series of environmental assessments of coal cleaning processes conducted by EPA's Air and Energy Engineering Research Laboratory at Research Triangle Park, North Carolina. The assessment activities involved continuing iterative studies to (1) identify and characterize industrial process discharges, (2) evaluate pollution control and waste disposal options, (3) compare estimates for environmental loadings with applicable standards and projected environmental goals, and (4) prioritize potential pollution problems and control technology needs. The conclusion from the overview was that "PCC is an established technology which is now being adopted and applied as a pollution control method--but there's still much to learn". One significant fact about PCC is that 5 to 15 percent of

1-2

the available Btu's is lost with the refuse. Although much of this Btu value is present as low-grade carbonaceous material, high-grade coal fragments too small for recovery are also present. Indeed, use of the PCC system for reducing the pyritic sulfur and the ash-forming mineral content of coals has other limitations which are subjects of research and development programs conducted by the U.S. EPA, the Department of Energy, and others. One of these areas deals with reducing the coal losses associated with coal cleaning and the ways to recover or otherwise utilize the high-grade coal rejected by the cleaning process [1].

The origin of the waste streams containing coal rejected by the PCC processes designed for maximum removal of pyritic sulfur and other ash-forming mineral matter is given in the overview report [1]. Such coal cleaning processes usually treat three size ranges of coals--coarse (3 x 3/8 inch), fine (3/8 inch x 28 mesh), and ultrafine (28 mesh x 0)--in individual circuits suited to maximize Btu recovery and the rejection of mineral matter and pyritic sulfur (see Figure 1). These goals are known to be difficult to achieve simultaneously and "effective cleaning" of the coal involves a compromise between ash and sulfur removal and Btu recovery. Therefore coal contaminated rejects are found in the waste streams from the fine coal and ultrafine coal circuits which, when combined with other wastewater discharged from the coal cleaning plant, make up a major portion of the wastewater from the process that must be treated before reuse or discharge. Examples of the other streams are slimes from desliming screens and the mechanical dewatering of clean coarse coal and fine coal dewatering. The solid wastes recovered from the wastewater treatment operations consist of extremely fine size materials and considerable amounts of water--a combination which must be disposed of with great care. In most operations, the water discharged from a coal cleaning plant goes directly to one or more ponds where the suspended solids settle out. Many plants incorporate thickeners in the process to treat the water for recycle or discharge to settling ponds. As a result of stream pollution control and the desire of the coal industry to improve fine coal recovery, recirculation and treatment of wash water are incorporated into modern coal cleaning plants. Such arrangements allow for closed water circuits, but closing the circuit results in buildup of slimes and it is

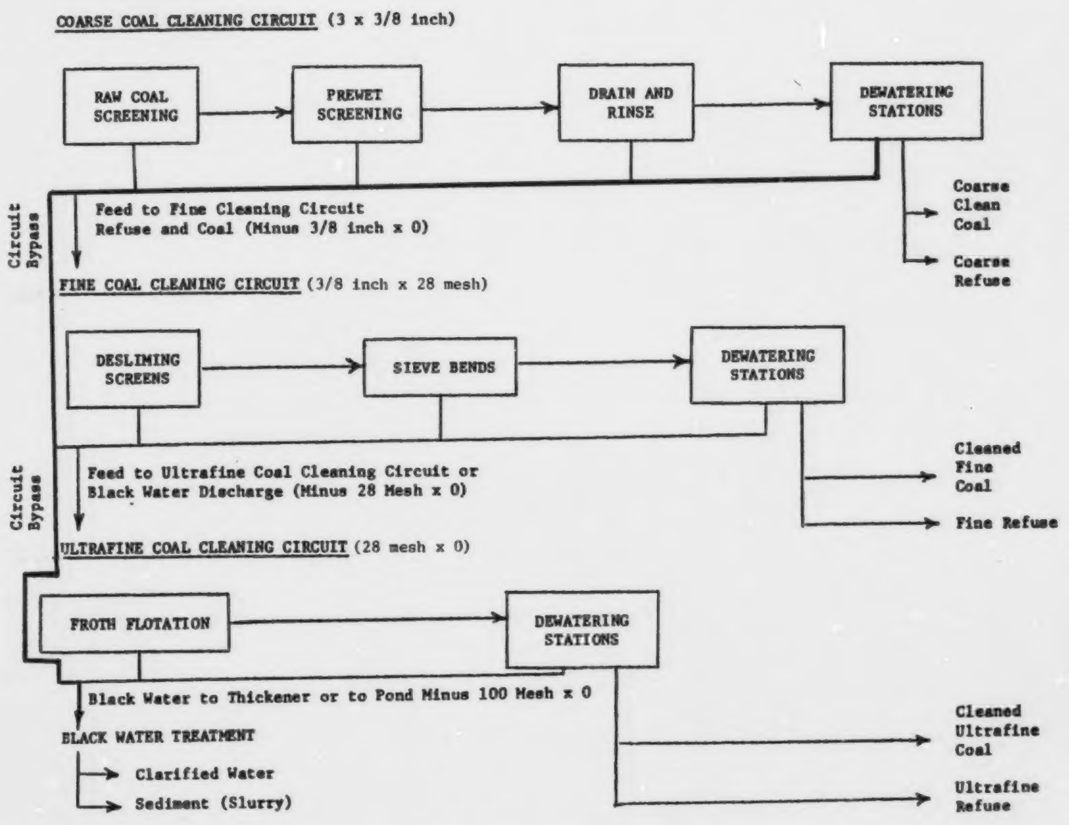
COARSE COAL CLEANING CIRCUIT (3 x 3/8 inch)
RAW COAL SCREENING
PREWET SCREENING
DRAIN AND RINSE
DEWATERING STATIONS
Coarse Clean Coal
Coarse Refuse
Circuit Bypass
Feed to Fine Cleaning Circuit
Refuse and Coal (Minus 3/8 inch x 0)
FINE COAL CLEANING CIRCUIT (3/8 inch x 28 mesh)
DESLIMING SCREENS
SIEVE BENDS
DEWATERING STATIONS
Cleaned Fine Coal
Fine Refuse
Circuit Bypass
Feed to Ultrafine Coal Cleaning Circuit or
Black Water Discharge (Minus 28 Mesh x 0)
ULTRAFINE COAL CLEANING CIRCUIT (28 mesh x 0)
FROTH FLOTATION
DEWATERING STATIONS
Cleaned Ultrafine Coal
Ultrafine Refuse
Black Water to Thickener or to Pond Minus 100 Mesh x 0
BLACK WATER TREATMENT
Clarified Water
Sediment (Slurry)

FIGURE 1. SUGGESTED SOURCES OF BLACK WATER FROM PHYSICAL COAL CLEANING PLANTS (Adapted from References 1 and 2)

necessary to remove a portion of the fine solids making up the slimes. Thickeners, cyclones, filters, and/or solid bowl centrifuges are used for this purpose. However, the recovered refuse contains coal and the recovered water often must be treated further (e.g., settling pond).

The study described in this report was initiated by U.S. EPA, IERL at Research Triangle Park as part of the overall assessment program with the aim of determining the applicability of the oil agglomeration technique for effluent and solid waste control from physical coal cleaning processes. The technique has the potential of recovering coal from rejects and producing a solid waste stream made up of finely divided mineral matter free of any significant coal values. In addition, if during an effluent treatment a separation of the liberated pyritic sulfur from the coal can be realized, a significant improvement in the recovered coal quality might be realized. This program also undertook a study of enhancing removal of the liberated pyrite present in the ultrafine and fine rejects over that possible using conventional oil agglomeration conditions. It is a continuation of an earlier EPA feasibility program [3].

BACKGROUND

Water immiscible liquids, usually hydrocarbons, have been used to separate coal from associated mineral matter [4]. In principle, such separation is an extension of selective wetting and association. That is, a process in which the hydrocarbons wet the hydrophobic surface of coal and associate with the oil phase, and the mineral matter, mostly hydrophilic, remains in aqueous suspension. Separation of the two phases takes place after agglomeration or coalescence occurs and produces agglomerates of clean coal wetted by the oil and an aqueous suspension of the mineral matter nearly free of combustible material. Effective separation can be made with coal smaller than 0.590 mm (minus 28 mesh) and often with coal too small for any other recovery scheme. Hydrocarbon fluids such as kerosene and fuel oils have been found very effective in separating the mineral matter from finely divided coal suspended in an aqueous slurry (thus reducing ash). The selective agglomeration process is also attractive because after wet face mining, wet size reduction, and conventional coal preparation operations, the coal does not have to be dried because the agglomerated coal can be readily dewatered by mechanical action. Therefore, use of oil for agglomeration provides energy trade-offs between oil used to dewater coal and fuel used for drying coal.

The amount of mineral matter that can be removed from coal by oil agglomeration can be increased by reducing the size of the coal fed. In general, pyrites are also released from the coal during size reduction, but they are not readily separated from the coal by the agglomeration process [5]. The major limitation of oil agglomeration as a technique to remove pyritic sulfur contaminants from coal is that separation of the liberated pyrite from coal is poor. The separation is poor because the surface properties of pyrites are similar to those of coal and in some coals pyritic sulfur is uniformly disseminated in the coal and in the size range of 0.5 to 20 micrometers. Extensive size reduction to liberate pyrites prior to oil agglomeration has found only limited success in reducing the pyritic sulfur content of the coal. Nonetheless the process has the potential to remove the trace elements commonly associated with the mineral matter in coal.

To keep the pyrites from accumulating in the oil phase, their surface properties must be altered to make them hydrophilic so that they will accumulate in the aqueous phase. The common pyrite depressant reagents developed for metallurgical froth flotation separations were found to be only partially successful in altering their surface properties and enhancing pyrite removal from the coal during agglomeration [3,4]. Studies on Iowa coals by Min [5] suggest approaches that might be used to alter the pyrite in Iowa coals. He found that chemically pretreating pulverized coal in a manner designed to oxidize the surface of the pyrite (to render it hydrophilic) was more effective than applying pyrite depressants. The pretreatment involved sparging a slightly alkaline aqueous slurry of a coal with air at 80 C. (The best pretreatment for an Iowa coal was the use of 0.5 g each of Na_2CO_3 and $Ca(OH)_2$ with 100 g of coal in 500 ml H_2O for 30 minutes which resulted in about a 42 percent reduction in total sulfur upon agglomeration.) Min also reported on other physical treatment sequences for the desulfurization of Iowa coal [5]. The most effective treatment sequence involved chemical comminution, float and sink separations at a specific gravity of 1.6, grinding of the float fraction, and recovery of the fine-size coal by agglomeration. Coal recovery was 82 to 84 weight percent with a reduction in pyritic sulfur of 85 to 86 percent. Optimum conditions identified for the Iowa coal in the study might not be applicable to coals from other regions, such as Appalachia.

Other studies on the theoretical aspects of coal cleaning by oil agglomeration have been reported, as well as laboratory, bench, and pilot scale studies on ash removal and dewatering [6-8]. Capes, et al. [6] pointed out that the utility of oil agglomeration of coal fines increases as more mechanized mining of progressively poorer grades of coal develops. Increased percentages of fines end up in the waste streams from the coal preparation plants (as black water or slimes). Usually water is impounded for long-term settling before discharge or reuse.

Research organizations involved in developing oil agglomeration methods, such as the U.S. Bureau of Mines, Pittsburgh Energy Research Center (PERC), concluded that excellent recoveries of low ash coal were possible both in a laboratory unit and a 500 pound-per-hour pilot plant but that sulfur reduction was negligible [9,10]. Bergbau-Forshung, GMBH, of West Germany, has developed the OLIFLOC process for recovery of ultrafine coal (minus 230 mesh) in a 3 ton-per-hour pilot plant [11]. Two, 30 ton-per-hour plants were installed during 1977. The Central Fuel Research Institute of India has also investigated oil agglomeration of a 25 percent ash coal in order to optimize processing conditions [7]. This fine coal, which was not amenable to economical cyclone washing or froth flotation, could be upgraded by oil agglomeration. A 2 ton-per-hour agglomeration unit has been installed for upgrading a coal slurry from the Lodna washery of the Bharat Coking Coal, Ltd., in India [12]. In Australia, the Broken Hill Proprietary Company, Ltd., has published the results of their studies on the process parameters controlling selective agglomeration of raw coal slurry in their pilot facility [8]. They found that agglomerates produced in a continuous process are considerably larger than those obtained from batch experiments. In these reports, only a small amount of work was reported on improving pyrite removal during agglomeration of black water discharges from coal cleaning plants.

The study completed by Battelle's Columbus Laboratories under EPA Contract No. 68-02-2112 demonstrated that coal recoveries of 90 percent or greater are attainable from fine coal slurry wastes using the oil agglomeration technique [3]. These high levels of recovery are attainable from fresh black water sediments generated during coal cleaning; aged sediments accumulated in slurry ponds; and excavated, weathered, and partially dried slurry pond sediments. The coal was of good quality and had lower ash and sulfur content than the cleaned coal shipped from the mine.

With regard to the enhancement of pyrite removal during agglomeration, it was demonstrated experimentally that pretreatment with sodium metaphosphate will permit removal of 42 percent of the pyritic sulfur from freshly ground minus 48 mesh coal. This is the same removal rate as that obtained by float-sink analysis, suggesting removal of all the liberated pyrite in the ground coal. Equally good results were obtained when a coal slurry was treated with oxygen in the presence of sodium carbonate at 25 C. These same treatments could be applied to black water and slurry pond sediments.

These results, albeit for only one coal type, suggest that the oil agglomeration technique may be further developed as a method to control effluents from coal cleaning plants. The applicability of the technique to wastes from other coal cleaning plants processing different seams at varying levels of complexity had to be demonstrated. The current program was undertaken to assess the applicability of the technique to a wider range of coals and coal cleaning wastes.

The overall objective of this program is to:

- Determine if the ability of the oil agglomeration technique to separate finely divided coal from mineral components can be applied to the control of effluents from coal cleaning plants
- Assess the environmental and cost consequences of the use of such separations as an alternative to or in conjunction with existing effluent controls.

Specific objectives of the work performed in the current program are:

1. Explore pretreatment methods of making the pyrite particles in black water hydrophilic.
2. Perform pretreatment and oil agglomeration to determine the pyrite rejection and coal recovery from coal cleaning plant black water.

3. Assess the performance of the oil agglomeration technique as an alternative for effluent control from coal cleaning plants and make recommendations for further study.

SECTION 2

CONCLUSIONS

This study demonstrates the potential applicability of the oil agglomeration technique for the control of black water effluents from coal cleaning plants processing four different coals. Removal and recovery of the coal from each of the black waters produced aqueous suspensions of mineral matter that settled more rapidly than the original black water. The sediment recovered from agglomeration appears to be less prone to acid generation during aeration than the total black water sediment.

The ash and sulfur content of the coal recovered by agglomeration is less than that of the cleaned coal. The quality of the recovered coal can be improved by chemical treatment of the sediment before agglomeration. Sodium sulfide appears to be one of the better agents to use because of the simplicity of the treatment process. Such pretreatment of the sediment can reduce the pyrites by up to 50 percent in the recovered coal over that without pretreatment. Even greater reductions in pyrite and ash are realized after pretreatment when the amount of oil used for agglomeration is reduced from 10 to about 2 percent and a two-stage air-float separation is used to recover the agglomerated coal.

Findings in this study suggest the following environmental and conservation advantages of the modified agglomeration process for the separation of the coal from the mineral matter in black water effluents:

- The recovered coal is easily dewatered despite its fine size.
- Chemical treatment of sediments before agglomeration tends to reduce the pyrite content of the recovered coal.
- Black water from cleaning low sulfur coals that contain fine and uniformly dispersed pyrites is less responsive to pretreatment probably because the pyrites are not present as liberated particles.

- The quantity of oil used for agglomeration affects the amount of pyrites and ash recovered with coal. Smaller amounts favor lower pyrites and ash in the recovered coal as well as high yields of coal per unit weight of oil; however, total recoveries are diminished.
- Use of a vegetable oil for agglomeration gives coal yields and recoveries equally as good as kerosene over an oil concentration range of from 2 to 10 percent. The recovered coal is less dusty.

The problems inherent in recovering coal from aged and weathered black water sediments, and the very high recovery of coal from fresh black water, led to the conclusion that a process to control effluents from a coal cleaning plant should be designed to treat the discharge at the rate it is generated rather than to treat the sediments accumulated and weathered in ponds.

The results from the engineering analysis which considered unit processes that could be combined with oil agglomeration effectively and economically to treat black water discharges are:

- Oil-agglomeration techniques could be implemented using unit processes and equipment conventionally employed by municipal water and wastewater treatment plants and by the coal industry.
- The cost of the oil-agglomeration recovery of fine coal from coal preparation effluent streams would approximate \$18 to \$22 per ton of coal recovered, assuming an oil price of \$.90 per gallon.

SECTION 3

RECOMMENDATIONS

It is recommended, based on the results of this study, that the oil agglomeration technique continue to be developed as a method to control effluents from coal cleaning plants and recover the energy value they contain. Additional work is needed on samples of black water from processing plants in other regions and/or seams such as the western coals. This work could be done in close cooperation with the current EPA program on the Environmental Assessment of Physical Coal Cleaning Processes. An experimental program is needed to establish process parameters and equipment needed to minimize the coal content in the coal cleaning plant effluent on a scale suitable for developing detailed process flow sheets and refining preliminary cost estimations for add-on or replacement effluent control systems. The data base should be increased sufficiently to permit detailed evaluation as to whether the technique should be considered on a pilot plant scale for on-site studies.

A study should also be initiated to determine not only the fate of the sediments resulting from the oil agglomeration process used to treat effluents from coal cleaning plants but also the impact of the successful chemical pretreatments on the pyrite loading of the sediments and their acid-generation potential.

SECTION 4

EXPERIMENTAL MATERIALS AND PROCEDURES

This section addresses the procedures used to obtain and characterize the black water samples and the various techniques used to treat the samples to maximize coal recovery and reduce liberated pyrite and ash-forming mineral carryover during oil agglomeration.

SAMPLE ACQUISITION AND HANDLING

Two samples used in this study were acquired by Versar, Inc., as part of their sampling campaigns at the Breckenridge coal cleaning plant near Morganfield, Union County, Kentucky (Kentucky No. 9 Seam) and the AMAX Delta coal cleaning in Williamson County, Illinois (Illinois No. 6 Seam). The third sample, obtained from McDowell County, West Virginia, was selected because of its low sulfur content compared to the two Versar samples. (Guidance for the selection was based on data from F. F. Aplan in Table 1 [13].)

Morganfield, Kentucky, Samples (Kentucky No. 9 Coal)

Samples from this coal cleaning plant were collected during a presampling site visit on July 24, 1979, while the facility was idle after processing Kentucky No. 9 coal and again on October 8-10, 1979, while it was actively processing Kentucky No. 9 coal. Our interpretation of the Breckenridge coal cleaning plant circuit provided by the TVA/Peabody Coal Company is given in Figure 2. Figures 2 and 3 show sampling sites; Figure 3 also depicts the layout of the facility. The black water (fine refuse slurry) is treated in a series of three settling ponds. Sediments in the first pond are rich in pyrite and easily supported a man walking on them. This suggests that the denser or coarse refuse in the slurry settles out quickly and the finer and less dense material (probably richer in coal) moves on to the other two ponds.

TABLE 1. POSSIBLE PHYSICAL COAL CLEANING PLANT SAMPLING SITES(a)

Sampling Site	Sample Identity	Location
1	Illinois Nos. 5 and 6	Jackson, Illinois
2	Pocahontas Nos. 3, 4, and 5	McDowell, West Virginia
3	Pittsburgh Seam	Washington, Pennsylvania
4	75 percent Pittsburgh 25 percent Lower Freeport	Harrison, Ohio

	Characteristics							
	Carbonaceous, percent			Ash Forming, percent			Total, percent	
Sampling Site	Ash	Sulfur	Weight	Ash	Sulfur	Weight	Ash	Sulfur
1	10.7	2.92	19.0	84.3	4.54	81	70.3	4.28
2	12.6	0.94	70.9	87.5	0.70	29.1	34.4	0.87
3	10.2	1.14	55.5	86.1	1.11	44.5	44.0	1.13
4	10.9	2.26	84.1	70.5	5.23	15.9	20.4	2.73

(a) Based on the listings in the report by F. F. Aplan.[13]

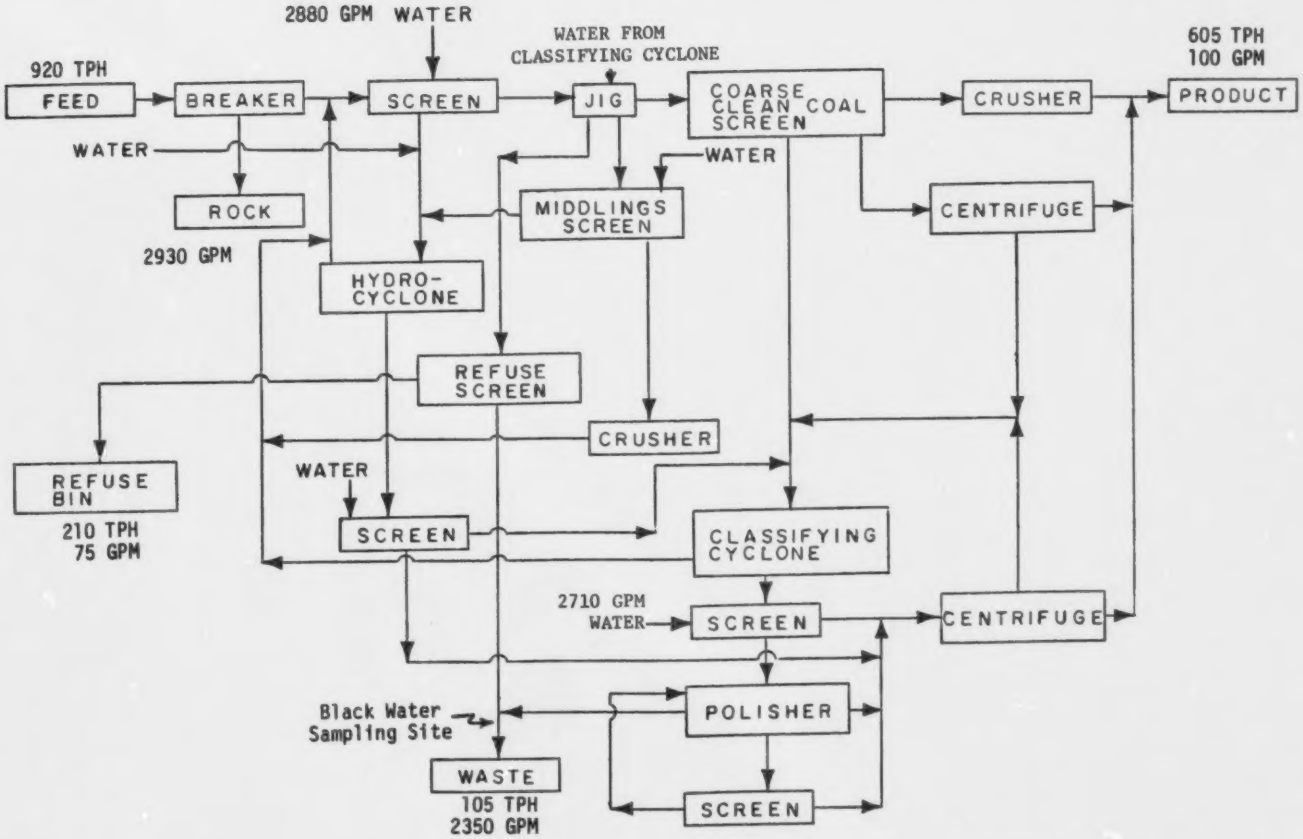
2880 GPM WATER
WATER FROM CLASSIFYING CYCLONE
605 TPH
100 GPM
920 TPH
FEED
BREAKER
SCREEN
JIG
COARSE CLEAN COAL SCREEN
CRUSHER
PRODUCT
WATER
WATER
ROCK
MIDDLINGS SCREEN
CENTRIFUGE
2930 GPM
HYDRO-CYCLONE
REFUSE SCREEN
CRUSHER
REFUSE BIN
WATER
SCREEN
210 TPH
75 GPM
CLASSIFYING CYCLONE
2710 GPM WATER
SCREEN
CENTRIFUGE
Black Water Sampling Site
POLISHER
WASTE
105 TPH
2350 GPM
SCREEN

FIGURE 2. SCHEMATIC OF THE COAL CLEANING CIRCUIT OF THE TVA/PEABODY COAL COMPANY BRECKENRIDGE COAL CLEANING PLANT AT MORGANFIELD, KENTUCKY

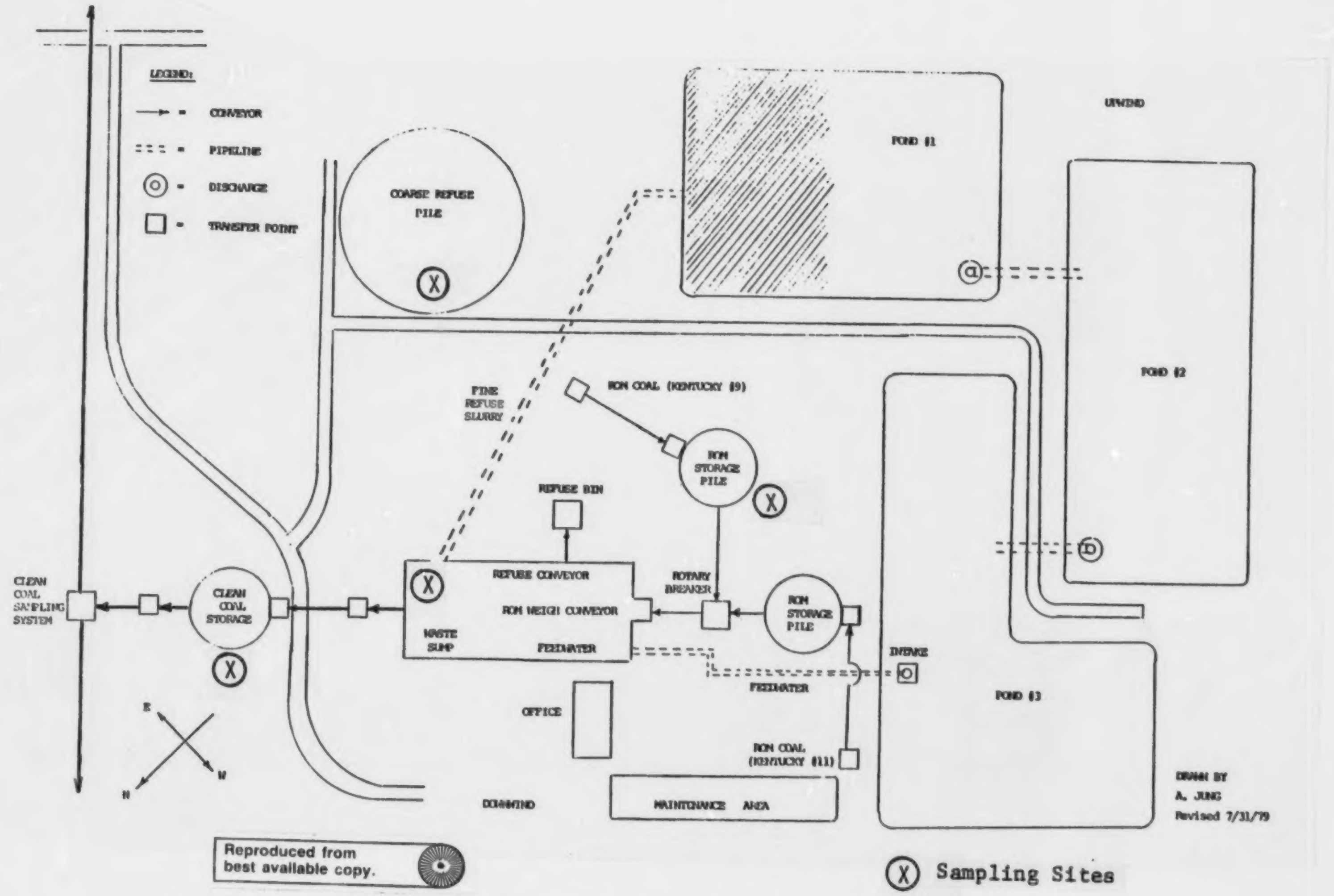
LEGEND:
CONVEYOR
PIPELINE
DISCHARGE
TRANSFER POINT
COARSE REFUSE PILE
POND #1
UPWIND
POND #2
POND #3
FINE REFUSE SLURRY
ROM COAL (KENTUCKY #9)
ROM STORAGE PILE
REFUSE BIN
REFUSE CONVEYOR
ROM WEIGH CONVEYOR
WASTE SUMP
FEEDWATER
ROTARY BREAKER
ROM STORAGE PILE
INTAKE
FEEDWATER
ROM COAL (KENTUCKY #11)
CLEAN COAL SAMPLING SYSTEM
CLEAN COAL STORAGE
OFFICE
MAINTENANCE AREA
DOWNWIND
E
W
N
a
b
DRAWN BY
A. JUNG
Revised 7/31/79
X Sampling Sites

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FIGURE 3. SAMPLING SITES FOR TVA/PEABODY COAL COMPANY BRECKENRIDGE COAL PREPARATION PLANT AT MORGANFIELD, KENTUCKY

Versar sampled raw coal, clean coal, coarse refuse, and black water from sampling sites shown in Figures 2 and 3. Samples were received at BCL within a two week period.

Williamson County, Illinois, Samples (Illinois No. 6 Coal)

Samples from the AMAX Delta coal cleaning plant processing Illinois No. 6 coal were collected by Versar Inc. on their second sampling excursion. The schematic of the coal cleaning circuit provided by AMAX is given in Figure 4. It differs from the previous coal cleaning circuit and incorporates a froth flotation to clean the fine and ultrafine coals. The noncoal fraction goes directly to the sump for fine slurry wastes. A sketch of the facility and the sites used by Versar, Inc. for sampling is given in Figure 5. The fine waste slurry is treated in desilting ponds and the clarified water is recycled.

Versar sampled raw coal, clean coal, coarse refuse and black water from the sampling sites shown in Figure 5. BCL received the samples within a two week period.

McDowell County, West Virginia, Samples (Pocahontas Nos. 3 and 4)

Samples from a coal cleaning plant processing coals from Pocahontas Seams Nos. 3 and 4 requested by BCL were sent to us directly from the plant. Although a schematic flow diagram of the plant was requested, none was received. However, it is known that the black water sample was obtained from an operating plant. The sample was taken from the underflow of a thickener used to pretreat the plant effluent before being discharged to a pond. In addition to the 5-gallon sample of black water, a sample of the cleaned metallurgical coal, the ROM coal feed, and some coarse refuse rich in pyrite were requested; only the refuse was not made available.

Belmont County, Ohio, Samples (Pittsburgh No. 8 Coal)

Samples from this coal cleaning plant were acquired during a previous EPA study (Contract No. 68-02-2112). The plant cleans deep-mined Pittsburgh Seam No. 8 coal. The schematic of the coal cleaning plant in Figure 6 shows the points where samples were taken. The samples taken were the ROM coal, the

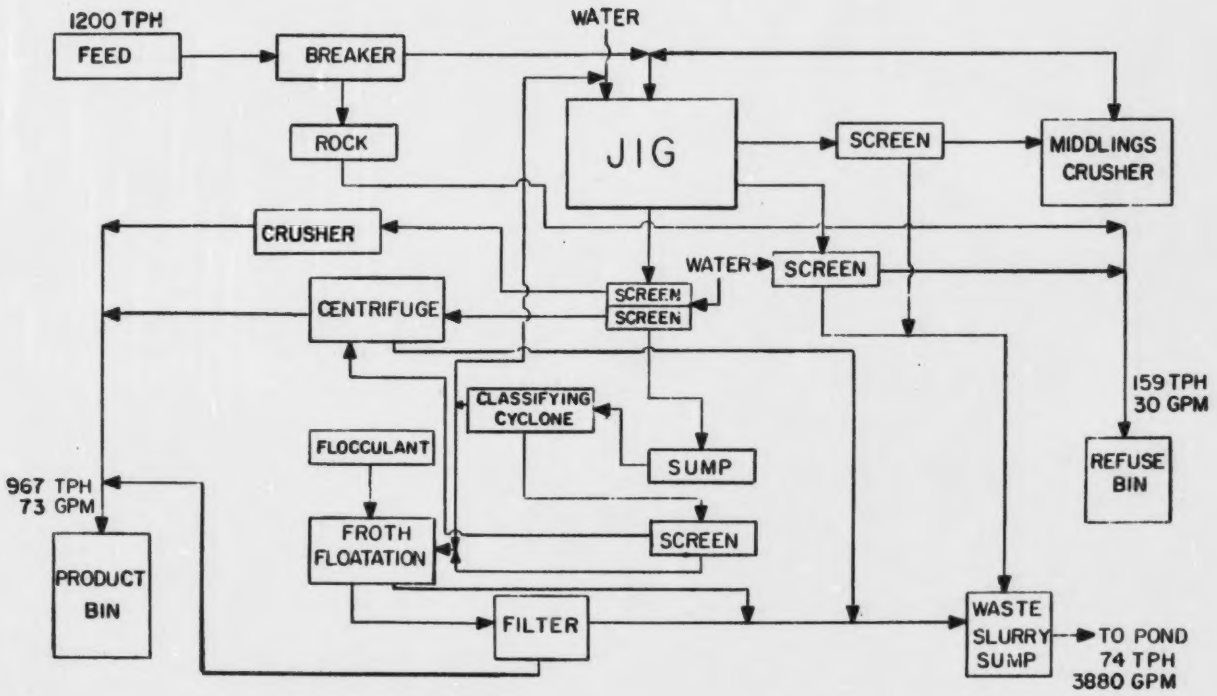
1200 TPH
FEED
BREAKER
WATER
ROCK
JIG
SCREEN
MIDDLINGS CRUSHER
CRUSHER
WATER
SCREEN
SCREEN
SCREEN
CENTRIFUGE
CLASSIFYING CYCLONE
SUMP
FLOCCULANT
FROTH FLOATATION
SCREEN
FILTER
967 TPH
73 GPM
PRODUCT BIN
159 TPH
30 GPM
REFUSE BIN
WASTE SLURRY SUMP
TO POND
74 TPH
3880 GPM

FIGURE 4. SCHEMATIC OF COAL CLEANING OPERATIONS AT THE DELTA COAL CLEANING PLANT, WILLIAMSON COUNTY, ILLINOIS

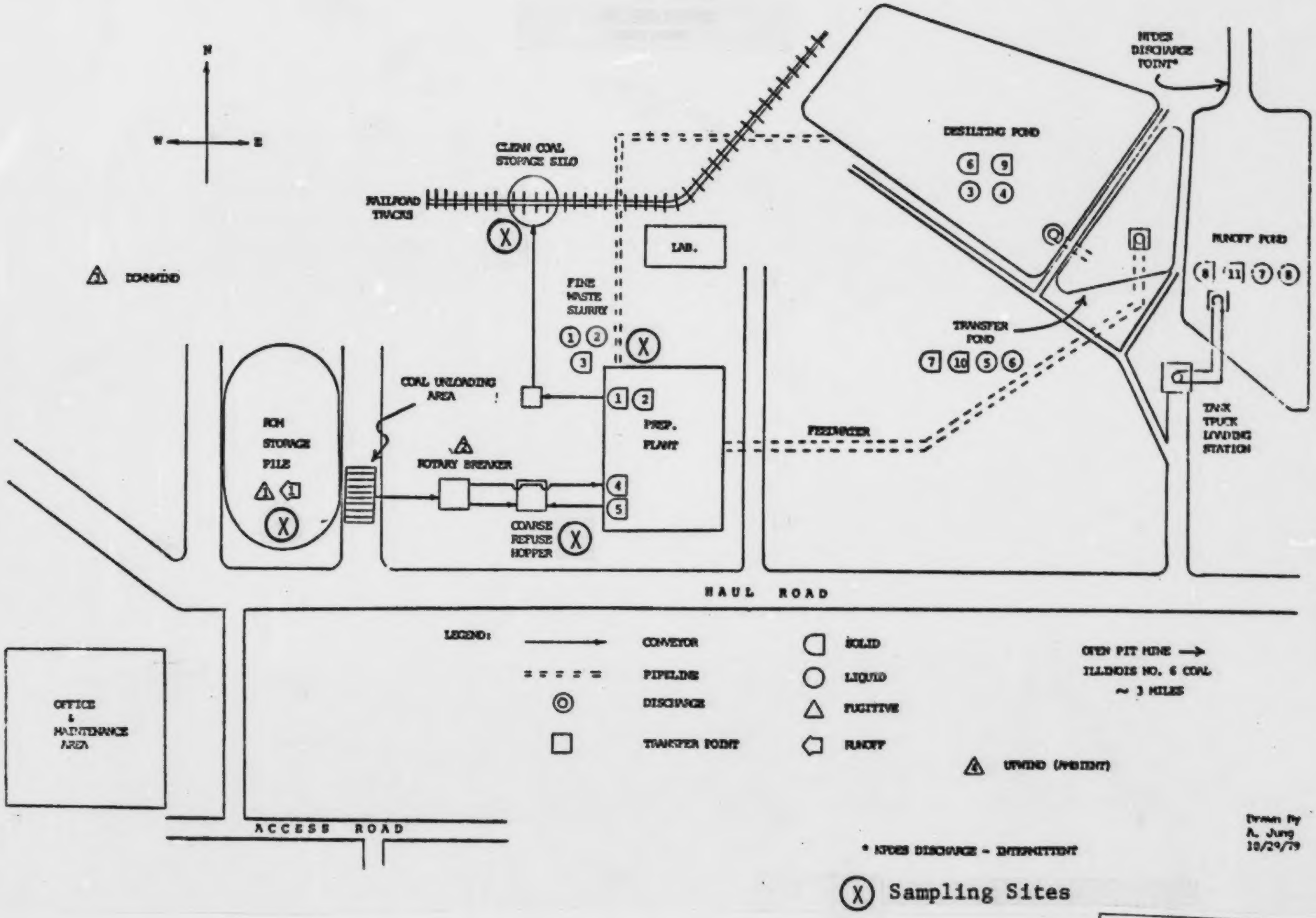
N
W
E
CLEAN COAL STORAGE SILO
RAILROAD TRACKS
DESILTING POND
NPDES DISCHARGE POINT*
RUNOFF POND
LAB.
FINE WASTE SLURRY
TRANSFER POND
COAL UNLOADING AREA
ROM STORAGE PILE
ROTARY BREAKER
PREP. PLANT
FEEDWATER
TANK TRUCK LOADING STATION
COARSE REFUSE HOPPER
HAUL ROAD
ACCESS ROAD
OFFICE & MAINTENANCE AREA
DOWNWIND
LEGEND:
CONVEYOR
PIPELINE
DISCHARGE
TRANSFER POINT
SOLID
LIQUID
FUGITIVE
RUNOFF
UPWIND (AMBIENT)
OPEN PIT MINE →
ILLINOIS NO. 6 COAL
~ 3 MILES
* NPDES DISCHARGE - INTERMITTENT
Drawn By
A. Jung
10/29/79
(X) Sampling Sites

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FIGURE 5. SAMPLING SITES OF THE DELTA COAL CLEANING PLANT, WILLIAMSON COUNTY, ILLINOIS (Illinois No. 6 Coal)

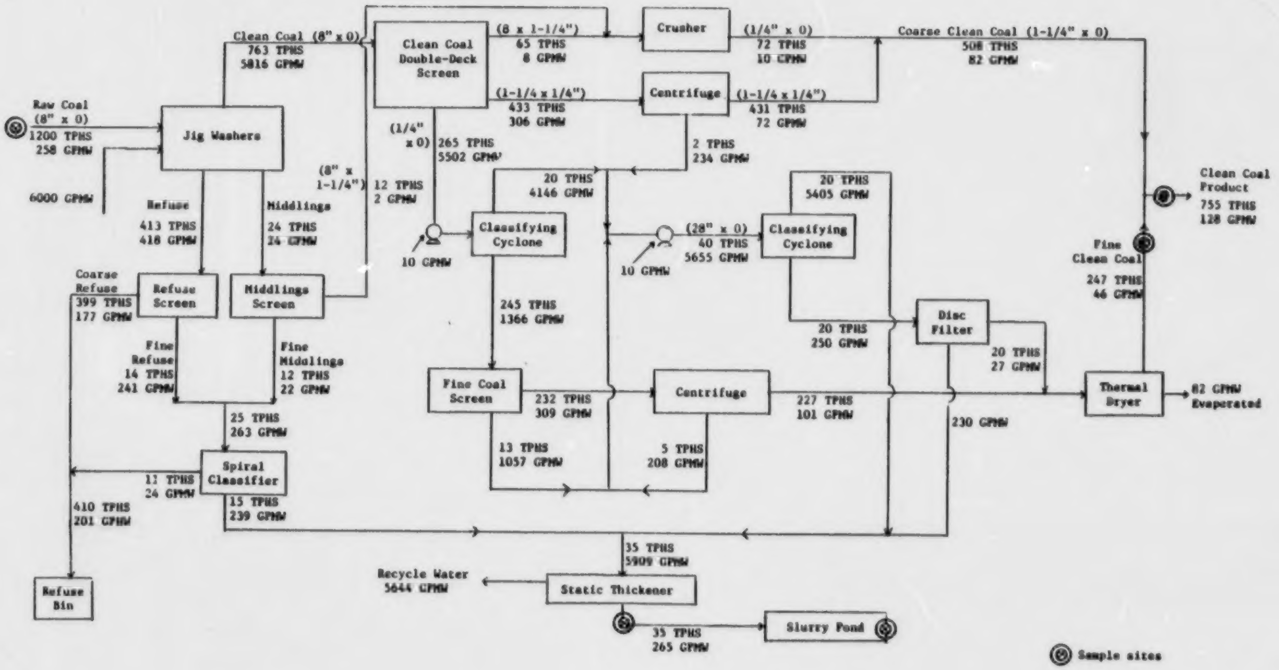

Raw Coal
(8" x 0)
1200 TPHS
258 GPMW
6000 GPMW
Jig Washers
Clean Coal (8" x 0)
763 TPHS
5816 GPMW
Refuse
413 TPHS
418 GPMW
Middlings
24 TPHS
24 GPMW
Coarse Refuse
399 TPHS
177 GPMW
Refuse Screen
Middlings Screen
Fine Refuse
14 TPHS
241 GPMW
Fine Midlings
12 TPHS
22 GPMW
25 TPHS
263 GPMW
Spiral Classifier
11 TPHS
24 GPMW
15 TPHS
239 GPMW
410 TPHS
201 GPMW
Refuse Bin
(8" x 1-1/4")
12 TPHS
2 GPMW
Clean Coal Double-Deck Screen
(8 x 1-1/4")
65 TPHS
8 GPMW
(1-1/4 x 1/4")
433 TPHS
306 GPMW
(1/4" x 0)
265 TPHS
5502 GPMW
10 GPMW
Classifying Cyclone
20 TPHS
4146 GPMW
245 TPHS
1366 GPMW
Fine Coal Screen
232 TPHS
309 GPMW
13 TPHS
1057 GPMW
Crusher
(1/4" x 0)
72 TPHS
10 GPMW
Centrifuge
(1-1/4 x 1/4")
431 TPHS
72 GPMW
2 TPHS
234 GPMW
10 GPMW
(28" x 0)
40 TPHS
5655 GPMW
Classifying Cyclone
20 TPHS
5405 GPMW
20 TPHS
250 GPMW
Centrifuge
227 TPHS
101 GPMW
5 TPHS
208 GPMW
Disc Filter
20 TPHS
27 GPMW
230 GPMW
Coarse Clean Coal (1-1/4" x 0)
508 TPHS
82 GPMW
Clean Coal Product
755 TPHS
128 GPMW
Fine Clean Coal
247 TPHS
46 GPMW
Thermal Dryer
82 GPMW Evaporated
35 TPHS
5909 GPMW
Recycle Water
5644 GPMW
Static Thickener
35 TPHS
265 GPMW
Slurry Pond
Sample sites

FIGURE 6. FLOW DIAGRAM OF THE NACCO MINING COMPANY COAL PREPARATION PLANT, BELMONT COUNTY, OHIO, IN 1977

cleaned coal ready for shipment, the dryer output, and the underflow of the static thickener (black water sample) which discharged into a slurry pond for long-term settling.

This sample which had been acquired in 1977 had been stored in a capped jar under a blanket of nitrogen. This material was the only material available early in the program for performing experiments, until the first sample was supplied by Versar, Inc.

THE OIL AGGLOMERATION PROCEDURE

The laboratory method used by Capes, et al. [6] for the classical oil agglomeration experiments have also been used by Min [5] and Mezey, et al. [3] in their initial experiments (i.e., separation of the agglomerated coal from the mineral suspension on a screen). The results from the classical laboratory method were found to be too dependent on variations in the laboratory worker's technique.

The procedure used here, however, is less subject to variations in technique and is an adaptation of the procedure developed by Min [5] and later used by Mezey, et al. [3]. It incorporates an air-float separation of the agglomerated coal from the aqueous suspension of the mineral matter and a resuspension of the separated coal in clean water followed by a second air float step. To assure at least 90 percent recovery of a coal with an ash content lower than the shipped clean coal, an unweathered black water sediment had to be used [3].

The standard oil agglomeration procedure used in this study consists of the following steps:

1. A weighed sample of the wet sediment (usually 25 g solid on a dry basis) was mixed with enough water to give the selected slurry concentration (usually 10 and 15 percent) and then agitated in the blender for a short interval to give a slurry.
2. An amount of oil was weighed out in a syringe to give the oil concentration desired based on the dry solids present and then added to the water coal slurry.
3. The mixture was blended for a period of time, usually 2.5 minutes.

4. After blending, the mixture was allowed to stand for about 15 to 30 seconds and then the entire mixture was poured (and washed) into an air-float cell (60 mm I.D. x 300 mm tube fitted with a fritted glass disc at the bottom). A slow flow of air bubbles lifted the agglomerated coal to the top of the aqueous phase where it was removed from the cell with a 48 (Tyler) mesh screen fashioned into a scoop.
5. Once all of the agglomerated coal fraction had been collected, it was resuspended in sufficient clean water to disperse the agglomerates, air floated a second time, and recovered.
6. The agglomerated coal was dried at 60 C in a vacuum oven and the water was evaporated from the mineral slurry in air in an oven set at 110 C. The recovered coal and mineral residue were weighed. The coal was analyzed, usually for ash and the sulfur types.

Modifications of this procedure were incorporated into the study and are identified in the discussions of the experiments where they were made. For example, when the effect of pH was examined, the pH of the slurry was changed by adding sulfuric acid or sodium hydroxide. The pH measured before the addition of the oil was taken as the pH of the agglomeration. Chemical pretreatment of the sediment was done at the desired agglomeration slurry concentration (usually 10 or 15 percent) and preceded the addition of the oil used in the agglomeration.

PRETREATMENT METHOD FOR THE ENHANCEMENT OF PYRITE REMOVAL

Because the surface properties of freshly exposed pyrite and coal are both classed as hydrophobic, the technique of oil agglomeration cannot separate the fine liberated pyrite present in the sediment from the coal. Unless some kind of treatment is used to alter the properties of the surface of the pyrite to make it wettable by water, it will report with the coal to the oil phase and be separated from the other mineral matter suspended in the aqueous phase. Any treatment that would selectively alter the surface of pyrite and not the coal would reverse this trend and enhance separation of the liberated pyrite present in the ultrafine coal fraction or discharged black water.

Potential treatment techniques were screened using finely divided pyrites (minus 100 mesh) prepared by grinding coarse pyrite specimens selected from the coarse refuse gathered at the site along with the coal and slurry samples. The hand-picked pieces of pyrite-rich material were first ground to a powder in a grinding mill. The pyrites were separated from the lighter materials by a "panning" technique using water in evaporating dishes. Pyrites separated in this manner were stored under water and an atmosphere of nitrogen in capped bottles until they were used in the pretreatment screening tests to reduce the oxidation of the freshly exposed surface of the pyrite. Although morphologies of FeS_2 other than pyrites exist in coal, these samples were believed to be adequate for the qualitative tests for which they were intended.

The pretreatment screening tests were designed to determine (after exposure of the fresh, finely ground pyrite to treatments) whether pyrite remained in the water phase after shaking with oil. Treatment was either with known pyrite depressing agents, oxidizing agents, or exposure to microwave energy. Chemicals used in the tests are given in the experimental results for each sediment. The screening tests consisted of the following steps:

1. A small amount of wet pyrite was placed in a test tube.
2. About 3 ml of water was added.
3. Treatment was provided by agitation or shaking.
4. The pH was adjusted with NaOH or H_2SO_4 solutions to the level used for agglomeration if desired.
5. Sufficient kerosene to give a well-defined layer was added and shaken vigorously.
6. The contents were allowed to stand until layers formed (2 to 3 minutes) and the observations on the distribution of the pyrite in the aqueous and kerosene layers were then recorded.

Parallel tests were run with finely ground clean coal samples using only the treatments that caused the pyrite to become wetted by water. These tests were conducted to assure that the hydrophobicity of the coal was not affected.

The most promising treatment and conditions were chosen for use with the black water sediments to determine if the technique would work on the waste stream mixtures produced during physical coal cleaning. The impact of the use of a froth flotation circuit on the surface characteristics of the coal and pyrite would be of importance because conditioning or flocculation agents are often used in the ultrafine coal cleaning circuit or in the thickener to improve separations.

In order to determine the effects of selected process variations of the oil agglomeration technique or the effects of chemical pretreatment of the sediment on the quality of coal recovered, a point of reference had to be established for each of the sediments used in this study. To do this, the results of the oil agglomeration of a slurry using a 10 percent oil concentration (based on the solids weight) following the standard oil agglomeration-air float procedure were considered to be baseline results on which comparisons could be made. Coal recovery is one evaluation criteria and is calculated using the following equation:

$$\text{Percentage Coal Recovery} = \frac{(100 - \%\ \text{Ash})_{\text{product}} \times (\%\ \text{of Feed})_{\text{product}}}{(100 - \%\ \text{Ash})_{\text{feed}}} \times 100$$

The product is the recovered coal, while the feed is the solids content of the sediment; the recovered coal product is given as the percentage of the solids in the feed slurry.

Another aspect of the study which needed baseline data was the reduction of the pyrites on a moisture-free and ash-free basis that occurs during the standard agglomeration-air float treatment. This was necessary to determine the effect that pretreatment of the sediment or changes in the agglomeration conditions have on the pyrites remaining in the recovered coal. The percentage reduction in the pyritic sulfur was determined using the equation

$$\begin{array}{c}\text{Percentage}\\ \text{Reduction}\\ \text{in Pyritic}\\ \text{Sulfur}\end{array} = \frac{\begin{pmatrix}\%\ S_{\text{pyr}}\ \text{without}\\ \text{Pretreatment}\end{pmatrix}_{\text{coal}} - \begin{pmatrix}\%\ S_{\text{pyr}}\ \text{with}\\ \text{Treatment}\end{pmatrix}_{\text{coal}}}{(\%\ S_{\text{pyr}}\ \text{without Pretreatment})_{\text{coal}}} \times 100$$

To estimate the amount of reduction in pyritic sulfur occuring during the standard agglomeration-air float procedure (i.e., the difference between the pyritic sulfur in the sediment and that in the recovered coal), the same equation was used substituting the percent S_{pyr} in the sediment for the untreated coal and the percent S_{pyr} in the recovered coal both on a moisture- and ash-free basis.

SECTION 5

RESULTS AND DISCUSSIONS

Results of the experiments with each of the black water sediments cover the characteristics of the black water, the coal recoveries realized by oil agglomeration, the characteristics of the residual mineral suspension, attempts at enhancing pyrite separation, and other observations.

BLACK WATER SEDIMENT CHARACTERIZATIONS

Each black water sample is discussed in the order of receipt except for the one from Belmont County, Ohio, acquired in 1977, which is discussed last.

Morganfield, Kentucky, Sample (Kentucky No. 9 Coal)

The wet slurry samples from the coal cleaning plant at Morganfield, Kentucky, received from Versar, Inc. on October 25, 1979, were allowed to settle for a period of about 3 days after which the solids and liquid levels were measured to estimate sediment and supernatant volumes. After the supernatant water was removed, the sediment was weighed to determine its specific gravity. These data and solids content of the slurry are given in Table 2 along with the moisture-free (MF) ash content and sulfur on a moisture-free and ash-free basis (MAF). The supernatant water had a pH of 6.8. Table 2 also presents the results of the wet sieve analysis of the sediment and the ash (MF) and total sulfur (MAF) of each of the isolated fractions. (Ashing was done at 750 C for a minimum of 2 hr and sulfur values were determined by the ESCHKA method.)

The data in Table 2 suggest that the sediment contains coarse (> 28 mesh) and fine (minus 200 mesh) material with significant amounts of ash-forming mineral matter. The sulfur and ash values follow each other in the fractions, with 35 x 100 mesh fractions having the lowest values for both. About 62 percent of the material was minus 100 mesh. This material contained 87 percent of the ash in the black water sediment.

TABLE 2. CHARACTERISTICS OF BLACK WATER FROM BRECKENRIDGE COAL CLEANING PLANT, MORGANFIELD, KENTUCKY KENTUCKY NO. 9 COAL

Composite after a 3-day period:

Sediment = 35.5 volume percent
Clear water = 64.5 volume percent
Density = 1.14 g/cc
pH = 7.1

Sediment after a 3-day period:

Density = -1.41 g/cc
Solids = 61.9 percent
Ash = 45.6 percent, MF(a)
S_{total} = 4.39 percent, MAF(b)
$S_{pyritic}$ = 2.88 percent, MAF

Wet Sieve Analysis

	Direct Data, %			Cumulative Data, %	
Tyler Mesh	Weight	Ash (MF)	S_{tot} (MAF)	Weight	Ash (MF)
+28	9.0	25.8	5.30	9.0	25.8
28 x 35	3.4	10.7	3.80	12.4	21.7
35 x 48	7.7	7.87	2.98	20.1	16.4
48 x 65	8.7	6.64	2.88	28.8	13.4
65 x 100	9.6	8.81	3.13	38.4	12.3
100 x 200	13.2	15.3	3.85	51.6	13.1
200 x 325	7.2	31.6	7.46	58.8	15.3
-325	41.2	67.6	NA(c)	100.0	36.9(d)

(61.6 percent minus 100 mesh)

(a) MF = moisture free basis.

(b) MAF = Moisture and ash free basis.

(c) NA = Not analyzed.

(d) Discrepancy with ash value of sediment may be caused by differences in the size of fractions used for analysis and their preparation for analysis.

Values for the run-of-mine (ROM) and the washed coal are compared to the black water sediment below:

		Sulfur, % (MAF)	
	Ash, % MF	Pyritic	Total
ROM Coal(a)	23.0	--	5.70
Cleaned Coal(a)	11.2	--	4.24
Black Water Sediment	45.6	2.88	4.39

(a) Values from an average of four samples received at Cumberland Steam Plant, TVA.

The values for ash and sulfur in the 35 x 100 mesh fractions are lower in ash and total sulfur than the shipped coal. Recovery of such a fraction with the washed coal would enhance the quality of the coal being shipped, providing it did not contribute a dust problem during handling and shipping.

The characteristics of the sediments collected from the sump and No. 1 pond while the plant at Morganfield, Kentucky was idle are shown in Table 3. The pond sediment collected near the outlet of the plant discharge is almost 75 percent ash* and 89 percent plus 100 mesh. Particles of pyrite were evident throughout the mass. The sediment from the sump, on the other hand, was 93 percent minus 100 mesh and had an average ash content of about 50 percent. The isolated sieve fractions were not analyzed for ash and sulfur because the samples were not considered to be representative of the discharge from an operating plant. The data does indicate the segregation that occurs in the settling ponds after the plant had been idle for about 3 weeks.

Williamson County, Illinois, Sample (Illinois No. 6)

The results of the characterization of the black water from the Williamson County, Illinois, coal cleaning plant received from Versar, Inc., on December 11, 1979, are given in Table 4. The clear water over the sediment had a pH of 8.2. The sediment amounted to only about 4 volume percent after settling for about 3 days and its density was about 1.27 g/cc. The solids content of the

---

*Mineral matter that contains clays, carbonates, quartz, and pyrites will have an "ash content" usually ranging between 80 and 90 percent because upon heating to 750 C these minerals will lose weight as they release water, carbon dioxide, and sulfur dioxide. Quartz will remain unchanged.

TABLE 3. CHARACTERISTICS OF BLACK WATER AND POND SEDIMENTS FROM IDLE BRECKENRIDGE PLANT, MORGANFIELD, KENTUCKY: KENTUCKY NO. 9 COAL (Sample Taken 7/24/79)

Blackwater Sediment (from sump)

Sediment: Density, liquid = 0.97 g/cc
Solid = 1.55 g/cc
Liquid pH = 8.1

Sediment solids = 58.4 percent
Ash = 49.9 percent MF

Wet Sieve Analysis:

Tyler Mesh	Direct Data, % weight	Cumulative Data, %
+28	1.0	1.0
28 x 35	0.3	1.3
35 x 48	0.4	1.7
48 x 65	0.3	2.0
65 x 100	0.5	2.5
100 x 200	4.6	7.1
200 x 325	6.8	13.9
-325	86.1	100

(92.9 percent minus 100 mesh)

Pond Sediment

Sediment: Density = 2.39 g/cc
pH = 6.7
Sediment solids = 85.3 percent
Ash = 73.2 percent MF

Wet Sieve Analysis:

Tyler Mesh	Direct Data, % weight	Cumulative Data, %
+28	36.2	36.2
28 x 35	12.2	48.4
35 x 48	14.5	62.9
48 x 65	10.9	73.8
65 x 100	8.9	82.7
100 x 200	6.3	89.0
200 x 325	1.7	90.7
-325	9.3	100

(11 percent minus 100 mesh)

TABLE 4. CHARACTERIZATION OF BLACK WATER FROM THE COAL CLEANING PLANT IN WILLIAMSON COUNTY, ILLINOIS: ILLINOIS NO. 6 COAL

Composite after a 3-day period:
Sediment = 3.9 volume percent
Clear Water = 96.1 volume percent
Density = $\sim$1.0 g/cc
pH = 8.2(a)

Sediment after a 3-day period:
Density = 1.27 g/cc
Solids = 66.4 percent
Ash = 51.1 percent (MF)
S_{total} = 6.13 percent (MAF)
$S_{pyritic}$ = 5.07 percent (MAF)

Wet Sieve Analysis:

	Direct Data, %			Cumulative Data, %	
Tyler Mesh	Weight	Ash (MF)	S_{tot} (MAF)	Weight	Ash (MF)
+ 28	0.6	6.2	2.57	0.6	6.2
28 x 35	1.5	6.8	2.61	2.1	6.6
35 x 48	5.0	10.1	2.66	7.1	9.1
48 x 65	9.2	20.3	3.26	16.3	15.4
65 x 100	8.7	33.7	3.98	25.0	21.8
100 x 200	18.7	44.8	6.04	43.7	31.6
200 x 325	11.0	55.0	8.15	54.7	36.3
- 325	45.3	71.9	8.94	100.0	52.4

(75 percent minus 100 mesh)

(a) Oil and coal floated on surface of water of the as-received sample.

sediment was 66 percent and the dried solid had an ash value of 51 percent. The sieve analysis of the wet solids suggests that the sediment is about 75 percent minus 100 mesh. The total sulfur in each fraction gradually increases with decreasing particle size and parallels the increase in ash content. The minus 325 fraction, which was about 45 percent of the solids by weight, was 71.9 percent ash. The values for the ROM coal and the washed coal are compared to the black water sediment below:

	Percent		
	Ash (MF)	S_{pyr} (MAF)	S_{tot} (MAF)
ROM Coal	25.7	1.78	3.67
Cleaned Coal	11.7	1.26	2.54
Black Water Sediment	51.1	5.07	6.13

Based on the ash and sulfur analyses of the individual sieve fractions, only the plus 28 x 48 fractions would appear to give coal with sulfur and ash values comparable to the cleaned coal being shipped. Segregation of the mineral matter from the coal during wet screening does not occur abruptly as it did for the sediments from Kentucky No. 9 coal. Instead, the mineral matter makes up a larger part of the successively smaller sized fractions. In effect, the black water sediment smaller than 100 mesh contained 89 percent of the ash in the sediment.

McDowell County, West Virginia, Samples (Pocahontas Nos. 3 and 4)

The results of the characterization of the black water received from a coal cleaning plant in McDowell County, West Virginia, are given in Table 5. The sediment after settling for a period of 3 days was about 40 percent by volume. The sediment was 56.9 percent solids and had an ash content of 28.7 percent. The sulfur on a moisture- and ash-free basis was very low for a reject stream, but the relatively low ash suggests a significant loss of coal value. About 88 percent of the material was found to be minus 100 mesh (about 70 percent minus 325 mesh) and contained about 97 percent of the ash-forming mineral matter. Ash analyses of the individual fractions suggest that about 91 percent of sediment's ash-forming mineral content is in the minus 325 mesh fraction. Both the total sulfur and pyritic sulfur are uniformly distributed in each of the sieve fractions, suggesting a close association with the coal

TABLE 5. CHARACTERISTICS OF BLACK WATER FROM McDOWELL COUNTY, WEST VIRGINIA, COAL CLEANING PLANT: POCAHONTAS NOS. 3 AND 4

Composite:

Sediment = 40.1 volume percent
Clear Water = 59.9 volume percent
Density = 1.02 g/cc
pH = 7.7(a)

Sediment:

Density = 1.30 g/cc
Solids = 56.9 percent
Ash = 28.7 percent (MF)
S_{total} = 0.83 percent (MAF)
$S_{pyritic}$ = 0.24 percent (MAF)

Wet Sieve Analysis:

	Direct Data, %				Cumulative Data, %	
Tyler Mesh	Weight	Ash (MF)	S_{tot} (MAF)	S_{pyr} (MAF)	Weight	Ash (MF)
+28	1.0	5.94	0.67	0.19	1.0	5.94
28 x 35	1.4	5.43	0.75	0.27	2.4	5.64
35 x 48	2.4	6.95	0.70	0.28	4.7	6.43
48 x 65	2.8	8.44	0.70	0.30	7.5	7.18
65 x 100	4.3	9.06	0.78	0.30	11.8	7.87
100 x 200	10.9	8.74	0.75	0.23	22.7	8.29
200 x 325	7.5	10.4	0.81	0.34	30.2	8.81
-325	69.8	37.4	0.83	0.30	100.0	28.8

(88.2 percent minus 100 mesh)

(a) Oil and some coal floated on surface of water of the as-received sample.

in each fraction, because there is no significant increase in the sulfur content of the last two high ash fractions, i.e., 200 x 325 and minus 325.

The ROM coal and the clean coal are compared to the black water sediment below:

	Percent		
	Ash (MF)	S_{pyr} (MAF)	S_{tot} (MAF)
ROM Coal	26.7	0.24	0.78
Clean Coal	6.04	0.10	0.64
Black Water Sediment	28.5	0.28	0.87

Belmont County, Ohio, Samples (Pittsburgh No. 8)

The characteristics of the black water from a coal cleaning plant in Belmont County, Ohio, are given in Table 6. The sediment had a solids content of 41.7 percent after settling for about 7 days. The ash content of the sediment was 50.8 percent (moisture free). The sediment contained 4.73 percent total sulfur and 2.61 percent pyritic sulfur. About 89 percent of the sediment was minus 100 mesh (about 79 percent was minus 325 mesh) and contained about 87 percent of the ash-forming minerals. The ash content of the wet sieve fractions suggests that the 28 x 100 mesh size fraction was mostly coal, but the ash-forming minerals existed mostly in the minus 325 mesh fraction. No sulfur determinations were made on the fractions.

The ROM coal and cleaned coal are compared to the black water sediment below:

	Percent		
	Ash (MF)	S_{pyr} (MAF)	S_{tot} (MAF)
ROM Coal	10.5	2.24	5.25
Clean Coal	9.24	2.04	4.88
Black Water Sediment	50.8	2.61	4.73

The black water sediment was stored in a capped glass jar which had been purged with nitrogen before sealing. Because this sample had been collected in 1977 and all of the characterizations were done at that time, it was used to perform experiments during the period between the start of the program and the first sample supplied to us by Versar, Inc.

TABLE 6. CHARACTERISTICS OF BLACK WATER FROM BELMONT COUNTY OHIO, COAL CLEANING PLANT: PITTSBURGH NO. 8 COAL

Composite after a 7-day period:

Sediment = 64.6 volume percent
Clear Water = 35.4 volume percent
Density = Not determined
pH = 7.0

Sediment after a 7-day period:

Density = ~1.2 g/cc
Solids = 41.7 percent
Ash = 50.8 percent (MF)
S_{total} = 4.73 percent (MAF)
$S_{pyritic}$ = 2.61 percent (MAF)

Wet Sieve Analysis:

	Direct Data, %		Cumulative Data, %	
Tyler Mesh	Weight	MF Ash	Weight	MF Ash
+ 28	3.3	8.55	3.3	8.55
28 x 35	1.5	17.74	4.8	11.42
35 x 48	1.7	20.36	6.5	13.75
48 x 65	1.8	18.28	8.3	14.74
65 x 100	2.4	18.97	10.7	15.68
100 x 200	5.3	13.87	16.0	15.08
200 x 325	5.0	10.72	21.0	14.04
-325	79.0	53.30	100.0	49.78

(89 percent minus 100 mesh)

5-10

COAL RECOVERIES AND REJECTION OF LIBERATED PYRITES

Coal recovered from black water sediments by oil agglomeration carries with it some of the pyrite liberated during size reduction of the coal and subsequent cleaning steps. This occurs because the fresh surfaces of pyrite are hydrophobic and will accumulate in the oil phase or at an oil-water interface. Experiments performed on the sediments concerning treatment of the sediment to minimize or eliminate the tendency of pyrites to be agglomerated with the coal are discussed in this section.

Treatment of Morganfield, Kentucky, Sediments

The black water sediments collected on July 24, 1979, from the sump when the plant was idle were used for some preliminary experiments to establish base line data on the agglomeration techniques. (No coal could be recovered by oil agglomeration from the pond sediments collected at the same time.) The results of various coal recovery trials are given in Table 7. High recoveries of reasonably good quality coal were obtained from this very finely divided feed slurry when the agitation of the 15 percent slurry with oil (kerosene) using a high shear mixing (blender) was followed by an air-float separation of the agglomerated coal. Separation of the agglomerated coal on a 48 mesh (Tyler) screen after low shear agitation gave low recoveries and considerable ash remained with the coal in Experiment 1. High shear agitation improved recoveries and lowered the ash (Experiment 2). Air-floating the recovered coal a second time (Experiment 3) reduced the ash to about 8 percent from about 10 percent when it was floated only once (compare Experiments 4 and 5). If the oil concentration was reduced from 10 percent of the dry sediment charge weight, as in Experiment 3, to 4 or 6 percent (Experiments 4 and 6) slightly lower ash was obtained and the coal recoveries were reasonable. By dispersing the agglomerated coal again in the high shear mixer as was done in Experiment 6, the ash was lowered to 7.5 percent. In Experiments 7 and 8 the kerosene used was reduced to 2.4 and 1.6 percent, respectively. In Experiment 7, a high shear agitation of the sediment using a 2 percent oil concentration was followed with a second addition of kerosene to the residual mineral matter remaining in suspension (0.4 percent). Although good coal recovery was achieved, the ash in the coal remained high. In Experiment 8, equal

TABLE 7. COAL RECOVERIES BY AGGLOMERATION WITH KEROSENE OF BLACK WATER SEDIMENT FROM IDLE PLANT AT MORGANFIELD, KENTUCKY (Sample Taken July 24, 1980)

(All 15 Percent Slurry Agitated for 2.5 Minutes)

Experiment No.	Agglomeration Conditions			Coal Product, %		
	Agitation	Separation	Oil Conc, %[(a)]	In Feed[(d)]	MF Ash	Coal Recovered[(e)]
1	Low shear	Screened	10	41.0	20.3	76.5
2	High shear	Screened	10	51.6	12.8	89.8
3	High shear	Air float twice[(b)]	10	54.8	8.1	100.5
4	High shear	Air float twice[(b)]	4	45.6	7.9	83.8
5	High shear	Air float once	4	55.3	10.2	99.1
6	High shear twice	Air float twice[(b)]	6	54.9	7.5	101.4
7	High shear Sediment Residue	Air float once once[(c)]	2.4 (total) 2 0.4	57.8	11.8	101.8
8	High shear Sediment Residue	Air float once once[(c)]	1.6 (total) 0.8 0.8	52.7	13.3	91.2

(a) Oil concentration based on weight of moisture free sediment used.

(b) Product coal redispersed second time.

(c) Residue was redispersed, agitated with additional oil, and air floated second time.

(d) As percentage of total weight of sediment feed.

(e) As percentage of coal in total sediment feed.

increments of 0.8 percent of oil were used to first agglomerate the coal and then treat the mineral suspension a second time. Even higher ash values were obtained, suggesting that rejection of the mineral matter from the agglomerated coal is in part dependent upon the amount of oil used. These results also suggest that the concept of removing the agglomerated coal by the air-float technique (or at least washing the recovered coal a second time and recovering the coal by the air-float technique) removes much of the free mineral matter. The technique, therefore, provides the best chance of removing liberated pyrites if their surface properties are made hydrophilic by chemical treatment. These results also support the selection of the technique as the standard agglomeration technique in this study. The coal recovered by this technique has an ash content of 7.5 to 8.0 percent, lower than that of the shipped cleaned coal, 11.2 percent.

Preliminary Treatment Studies--

The black water sediment (from sump) collected July 24, 1979, was used in some preliminary pretreatment experiments to attempt to enhance the hydrophilicity of the liberated pyrite and thereby keep it in the aqueous phase during agglomeration. Before these experiments were completed, black water sediments more typical of the operating plant were to be obtained, so no effort was made to determine the effect on the sulfur values of this finely divided feed (86 percent minus 325 mesh).

The approach used in the experiments listed in Table 8 was to determine the effect of sodium metaphosphate treatment on the ash in the recovered coal. All the sediment samples (25 g dry weight) were treated after dilution to a 15 percent slurry concentration by adding either 0.25 to 0.50 g of a 5 percent by weight sodium metaphosphate solution. (This material was found to be effective in reducing the pyrite concentration of coal recovered from sediments originating from cleaning Pittsburgh No. 8 coal [3].) The mixture was agitated together in a high shear mixer for 2.5 minutes before adding the oil (kerosene) and agglomerating the coal. The agglomerated coal was air-floated from the mixture. The coal was agglomerated using a 2 percent oil concentration. The remaining oil (either 0.4 or 0.8 percent concentration) was used to air-float the remaining coal from the residue (i.e., the mineral matter suspension). In Experiments 9 and 10 the sodium metaphosphate treatment appears

TABLE 8. TREATMENT OF SEDIMENT FROM MORGANFIELD, KENTUCKY, WITH SODIUM METAPHOSPHATE SOLUTION FOR PYRITE REMOVAL (Sample Taken July 24, 1979 While Plant Idle)

(All 15 Percent Slurry Agitated for 2.5 Minutes)

Experiment No.	Agglomeration Conditions			Coal Product, percent(a)		g Coal Recovered / g Oil Used	Sodium Metaphosphate,(c) percentage of sediment dry weight
	Agitation	Separation	Oil Conc, %	Coal Recovery(b)	Ash (MF)		
	Standard agglomeration		10	102.5	8.1	5.56	—
9	High shear	Air float Coal once Residue once	2.4 overall 2.0 0.4	97.1	12.8	25.0	0.05
10	High shear	Air float Coal once Residue once	2.4 overall 2.0 0.4	98.0	16.3	25.0	0.10
11	High shear	Screen Coal once Residue once	2.4 overall 2.0 0.4	95.3	16.4	25.0	0.05
12	High shear twice	Air float Coal twice Residue once	2.4 overall 2.0 0.4	99.5	5.9	20	0.10

(a) Since these were with preliminary samples, sulfur was not determined.

(b) As a percent of coal in sediment.

(c) As a 5 weight percent sodium metaphosphate solution; treated for 2.5 minutes during high shear agitation.

to cause an increase in the ash content compared to Experiments 5, 7, and 8 given in Table 7. Use of a 48 mesh screen reduced the yield and did not lower the ash content (Experiment 11). However, when the recovered coal was redispersed by high shear mixing as it was in Experiment 12 and recovered by air flotation a second time, the ash content was reduced from 16 percent to 6 percent and the apparent coal recovery remained high. Again it must be remembered that this work was done using very fine sediment that could adhere to the agglomerated coal and was not representative of the black water sediment being discharged from an operating plant.

One striking feature of the experiments in Table 8 is that at these very low levels of oil concentration (i.e., 2.4 percent overall) about 20 to 25 g of coal was recovered per gram of oil used compared to the standard agglomeration yield of 5.6 g. These results suggest that similar experiments on the Illinois and West Virginia sediments should be considered. Any reduction in the oil requirements can greatly reduce the cost of coal recovery [3]. However, the greater yield found here results in part from the use of the finely divided sediment.

Treatment Screening Experiments--

Fresh pyrites picked from the coarse refuse obtained on October 25, 1979, were ground. The liberated pyritic material was separated as described in Section 4 and used in experiments to give a crude but useful test of the effect a treatment had on propensity of pyrite to report to the kerosene phase. Table 9 shows the results from experiments in which the effect of pH was determined, with and without a reagent present. The results suggest that at a pH of 8.5 more pyrite is wetted with water at least alone or with sodium metaphosphate present. Sodium sulfite has potential as a pyrite depressant (makes it hydrophilic) in mineral froth flotation operations. However it was not judged to be an effective pyrite depressant with coal pyrites.

Other treatments listed in Table 10 were tried. The most striking effects on the surface properties of pyrites were obtained by treatment with:

1. A 5 percent solution of sodium metaphosphate at 24 C and sparging with O_2
2. A 10 percent solution of sodium sulfide solution at 24 C

TABLE 9. EFFECT OF pH ON MORGANFIELD, KENTUCKY, PYRITE BEHAVIOR(a)

Agent(b)	pH(c)	Results(d)
None	7	I(W)
None	8.5	WI
Sodium metaphosphate $NaPO_3$	8.5	W(I)
Sodium metaphosphate $NaPO_3$	9.5	WI
None	9.0	WI
Sodium sulfite Na_2SO_3	7.5	WI
Sodium sulfite Na_2SO_3	8.5	WI

(a) All experiments at 24 C, about 10 percent slurry by weight.

(b) Agents usually as 0.5 ml of 5 percent solution (by weight).

(c) pH was adjusted using dilute solutions of H_2SO_4 and NaOH.

(d) Key:
(Best) W = pyrite almost completely in water phase
W(I) = pyrite largely in water phase, some in kerosene/water interface
WI = pyrite partly in water phase, partly in kerosene/water interface
(Worst) I(W) = pyrite largely in kerosene water interface, some in water phase

TABLE 10. EFFECT OF CHEMICAL PRETREATMENT ON MORGANFIELD, KENTUCKY, PYRITE BEHAVIOR(a)

Agent	Temp (C)	O_2 Sparge	Results(c)
None	24	Yes	I(W)
None	90	Yes	WI
Na_2CO_3	24	No	WI
Na_2CO_3	24	Yes	W(I)
Na_2CO_3	90	No	W(I)
Na_2CO_3	90	Yes	W
$NaPO_3$	24	Yes	W(I)
Na_2O_2(b)	24	No	W(I)
Na_2O_2(b)	24	Yes	W(I)
Na_2O_2	90	No	WI
Na_2S	24	Yes	W(I)
Na_2S	90	Yes	WI
Na_2S	90	No	WI
Na_2CO_3	Microwave for 2 min		W(I)
Na_2CO_3	Microwave for 2 min		W(I)
Na_2CO_3	Microwave for 2 min, add $NaPO_3$		WI

(a) All tests done at pH = 8.5.

(b) Done with large amounts of Na_2O_2 (~1 ml 5 percent solution per 1g FeS_2 (dry basis).

(c) Key:

(Best) W = pyrite almost completely in water phase

W(I) = pyrite largely in water phase and some at kerosene/water interface

WI = pyrite partly in water phase, partly at kersosene/water interface

(Worst) I(W) = pyrite largely at kerosene/water interface and some in water phase.

3. A 5 percent solution of sodium peroxide and sparging with O_2 at 24 C
4. A 10 percent sodium carbonate solution and sparging with O_2 at 90 C.

It was judged that although microwave heating (2450 MHz, 500 watts) for 2 minutes in the presence of sodium carbonate did alter the pyrite distribution between water and kerosene, similar effects could be achieved at room temperature using other reagents. With all of the water present, it would appear that microwaves have little direct effect on the pyrites but are just heating the water.

Parallel treatment tests on freshly ground cleaned coal from Morganfield, Kentucky, suggested that the behavior of the coal was not altered during treatment since after the treatment and mixing with kerosene all the coal was found in the kerosene phase.

Sediment Treatment Experiments

Based on the results from preliminary experiments with fine black water and the preceding treatment screening tests, the following pretreatment sequences were used prior to performing a standard oil agglomeration on the black water sediment slurries:

Treatment Sequence	Conditions
1	Simple agglomeration (no pretreatment), pH = 8.5
2	4 g of 5 percent solution of $NaPO_3$ per 100 g sediment (dry basis) as pretreatment, sparge 15 min with O_2 with pH = 8.5
3	2 g of 10 percent solution of Na_2S (as $Na_2S \cdot 9H_2O$) per 100 g sediment (dry basis), waiting 15 min after pH adjusted to 8.5
4	4 g of 5 percent solution of Na_2O_2 per 100 g sediment (dry basis), sparging for 15 min with O_2 at pH = 8.5
5	2 g of 10 percent solution of Na_2CO_3 per 100 g sediment (dry basis), sparging for 15 min with O_2 at pH = 8.5 while heating at 90 C

All experiments used 25 g of sediment on a dry basis and all agglomerations were performed at a pH = 8.5. Oxygen flow was about 800 ml/min.

The effectiveness of the treatment was measured by the reduction in the pyrite in the recovered coal compared to that obtained without any treatment (Number 1 above). The results in Table 11 suggest that the treatment with sodium sulfide (Number 3 above) has the most effect on the reduction of pyrite remaining in the coal. The recovered coal ash value was reduced to 5.2 percent and the pyritic sulfur value to 0.86 percent or a reduction of about 38 percent from the pyrite value in the coal recovered without pretreatment. The cleaned coal shipped from the plant had an ash content of 11.2 percent and 4.24 percent total sulfur (about one-half can be considered pyritic sulfur). All of the treated sediments yielded coals lower in pyritic sulfur.

The recovered coal after the other treatments was lower in ash and total sulfur than the shipped coal. The coal recoveries of 73.5 to 81.9 percent were lower than expected for an unweathered black water sediment, which was usually in excess of 90 percent. It is not known if the decline in recovery resulted from the treatment sequence affecting the coal, or from the action of air on the sediment during the two months between sample receipt and performance of the experiments. (Care was taken to purge the void space of the jar with nitrogen after each series of samples was removed.)

Some resolution of the question could be made from the data in Table 12, where the results of experiments in which sediment was used within three weeks after receiving the sample are given. The objective of this set of experiments was to determine the effectiveness of the sodium metaphosphate treatment (without sparging with O_2) on reduction of the pyrite in the coal recovered after oil agglomeration. The results suggest that the pyrite in the recovered coal was lowest without pretreatment and increased with the treatment sequence. Pretreatment made pyrite rejection worse. It should be noted that the ash values of the coal increased with the treatment to 7.65 percent, which is about the same as the 7.2 percent obtained for ash values in Table 11. However, the coal recoveries in Table 11 even with pretreatment are higher and closer to values usually obtained when unweathered black water sediment was used, which suggests that the surface characteristics of the coal in the sediment had changed when the later experiments were done. (Compare results in Table 9 at pH 8.5 to results from treatment sequence No. 1.)

TABLE 11. REDUCTION OF PYRITE IN COAL RECOVERED BY OIL AGGLOMERATION AFTER PRETREATMENT OF BLACK WATER SEDIMENT FROM MORGANFIELD, KENTUCKY (Sample Obtained October 25, 1979)[(a)]

Pretreatment Sequence	Pretreatment Condition	Recovered Coal Values, %				Percentage Reduction in Pyritic Sulfur	
		Recovery	Ash (MF)	S_{pyr} (MAF)	S_{tot} (MAF)	(b)	(c)
Black Water Sediment		--	45.6	2.88	4.39	--	--
Number 1	None	77.3	7.2	1.40	3.20	--	(51.4)
Number 2	$NaPO_3 + O_2$, 15 min	81.9	7.2	1.39	3.13	0.7	(51.7)
Number 3	Na_2S only	74.4	5.2	0.86	2.60	38.6	(70.1)
Number 4	$Na_2O_2 + O_2$, 15 min	74.5	6.4	1.19	2.98	15.0	(58.7)
Number 5	$Na_2CO_3 + O_2$, 15 min @ 90 C	73.5	6.2	1.14	2.91	18.6	(60.4)

(a) Experiments performed 12/26/79.

(b) Values based on pyrites in recovered coal when no reagent used.

(c) Values based on pyrites in sediment.

TABLE 12. REDUCTION OF PYRITE IN COAL RECOVERED BY OIL AGGLOMERATION AFTER TREATMENT OF BLACK WATER SEDIMENT FROM MORGANFIELD, KENTUCKY (Sample Obtained October 25, 1979)(a)

	Coal Recovery, %	Ash, % MF	Sulfur, % MAF		Percentage Reduction in Pyritic Sulfur	
			Pyritic	Total	(e)	(d)
Blackwater Sediment	--	45.6	2.88	4.39		--
Recovered Coal, no reagent	91.4(c)	6.78	1.34	3.28	--	(53.5)
at pH 8.5(b)	103.6(c)	7.29	1.60	3.44	-19.4	(44.4)
at pH 8.5 and sodium metaphosphate(b)	101.8(c)	7.65	1.76	3.51	-31.3	(38.9)

(a) Experiments performed 11/12/79.

(b) Treatments to improve pyrite separation included adjustment to pH 8.5 and the contact of a solution of sodium metaphosphate adjusting pH to 8.5 for 15 min (0.5g of 5 percent $NaPO_3$ solution to 100g coal).

(c) Values after drying at 60 C under 380 Torr vacuum for 2 hr.

(d) Values in parentheses based on pyrites in the sediment.

(e) Values based on pyrites in recovered coal when no reagent used (the negative value indicates an increase after treatment).

A comparison of the sulfur and ash values of coal recovered by agglomeration after pretreatment with the other samples of coal received from the Morganfield, Kentucky, coal cleaning plant is given in Table 13. The following conclusions are based on the data:

- There is only a modest reduction in sulfur but a significant reduction in ash during the cleaning of the coal.
- The coal recovered from black water by standard agglomeration is lower in ash than the cleaned coal shipped from the plant, but no reduction in pyrite occurs, suggesting that any liberated pyrite was isolated with the recovered coal.
- The coal recovered after pretreatment is significantly lower in ash and pyritic (and total) sulfur, suggesting that liberated pyritic sulfur is not recovered with the coal.

Treatment of Sediment from Williamson County, Illinois

Treatment Screening Experiments--

Fresh pyrites were picked from the coarse refuse furnished with black water samples received December 11, 1979. Screening experiments for the suppression of pyrite agglomeration were attempted but none of the freshly ground pyrites isolated from the coarse refuse sample or any pyrite isolated from the ROM coal would float in the kerosene phase. What appeared to be large, unweathered samples were ground to minus 100 mesh several times in an attempt to expose new surfaces for the test, but none of the material migrated to the kerosene-water interface. This behavior was not observed in any of the previous samples and suggests that weathered refuse had been collected.

Because the screening experiments could not be performed, it had to be assumed that the pyrites in the black water sediment were not weathered and that the reagents most effective on the pyrites from Kentucky No. 9 coal would also be effective on the pyrites from Illinois No. 6 coal. Therefore the same treatments were performed on the Illinois black water sediment as were used before on the Kentucky sediments.

Sediment Treatment Experiments--

The sequence of treatments outlined for the Kentucky No. 9 sediments was used for the Illinois No. 6 sediment using the same procedure as outlined in

TABLE 13. ANALYSIS OF COAL SAMPLES FROM MORGANFIELD, KENTUCKY, COAL CLEANING PLANT COMPARED WITH COALS RECOVERED BY AGGLOMERATION (All Samples Obtained October 24, 1979)

	Percent		
	Ash (MF)	S_{pyr} (MAF)	S_{tot} (MAF)
Run-of-Mine Coal	23.1	1.39	3.62(b)
TVA sample(a)	19.0	--	5.70
Clean Coal	11.2	1.50	6.24(b)
TVA sample	10.3	--	4.24
Black Water Sediment	45.6	2.88	4.39
Coal Recovered by Agglomeration			
No treatment	7.2	1.40	3.20
After treatment with sodium sulfide (Method 3)	5.2	0.86	2.60

(a) Values from an average of four samples received at TVA Cumberland Steam Plant.

(b) These values are obviously in error due to poor sampling; therefore, the TVA values should be considered more representative.

that section of the report. Sediment treatments were for 15 minutes, after which the pH was adjusted to 8.5 before performing a standard agglomeration using a 10 percent kerosene concentration.

The results of the pretreatment experiments given in Table 14 suggest that a treatment of the sediment at 24 C with a 10 percent (by weight) solution of sodium sulfide at a rate of 2 g of solution per 100 g of dry sediment weight is about as effective as sparging with O_2 in the presence of sodium peroxide or sparging with O_2 in the presence of sodium carbonate at 90 C.

A comparison of the sulfur and ash values for the coal recovered by agglomeration before and after pretreatment with the other samples of the Illinois No. 6 coal is made in Table 15. Based on those data, the following conclusions are made:

- The coal can be cleaned to lower levels of ash and pyritic sulfur than can be attained in coal recovered by standard oil agglomeration of the black water sediment.
- Liberated pyritic sulfur is agglomerated with the coal recovered from the black water.
- The coal recovered after pretreatment has ash and pyritic sulfur values at least as low as the shipped clean coal.

Reduced Oil Level Experiment--

The results from experiments with Kentucky sediments, in which the yield of coal per unit weight of oil used was significantly greater than when standard agglomeration was used, suggested that such experiments should be tried with the black water sediments from Williamson County, Illinois. In addition, the impact of such treatment on the pyritic sulfur content of the recovered coal had to be determined. The data from these experiments are compared to the results from standard agglomeration of sediments with and without treatment with sodium sulfide in Table 16. The results suggest that several effects occurred:

- The amount of coal recovered per gram of kerosene was a maximum of about 19 g coal when only 2 percent kerosene concentration was used. The recovered coal had the lowest ash of those recovered by this technique.

TABLE 14. REDUCTION OF PYRITE IN COAL RECOVERED BY OIL AGGLOMERATION AFTER PRETREATMENT OF BLACK WATER SEDIMENT FROM WILLIAMSON COUNTY, ILLINOIS

Pretreatment Sequence	Pretreatment Condition	Recovered Coal Values, %				Percentage Reduction in Pyritic Sulfur	
		Recovery	Ash (MF)	S_{pyr} (MAF)	S_{tot} (MAF)	(a)	(b)
Black Water Sediment			51.1	5.07	6.13	—	—
Number 1	None	85.6	10.4	2.32	3.72	--	(54.2)
Number 2	$NaPO_3 + O_2$, 15 min @ 25 C	81.4	10.3	2.26	3.68	2.6	(55.4)
Number 3	Na_2S only, 15 min @ 25 C	83.0	7.3	1.13	2.51	51.3	(78.9)
Number 4	$Na_2O_2 + O_2$, 15 min @ 25 C	82.9	7.4	1.20	2.49	48.3	(76.3)
Number 5	$Na_2CO_3 + O_2$, 15 min @ 90 C	81.5	7.3	1.14	2.39	50.9	(77.5)

(a) Values based on pyrites in coal recovered with no reagent used.

(b) Values based on pyrites in sediments.

TABLE 15. ANALYSIS OF COAL SAMPLES FROM WILLIAMSON COUNTY, ILLINOIS, COAL CLEANING PLANT COMPARED WITH COALS RECOVERED BY AGGLOMERATION

	Percent		
	Ash (MF)	S_{pyr} (MAF)	S_{tot} (MAF)
Run-of-Mine Coal	25.7	1.78	3.67
Cleaned Coal	11.7	1.26	2.55
Blackwater Sediment	51.1	5.07	6.13
Coal Recovered by Agglomeration			
No treatment	10.4	2.32	3.72
After treatment with sodium sulfide (Method 3)	7.3	1.13	2.51

TABLE 16. IMPACT ON PYRITIC SULFUR CONTENT OF COAL RECOVERED USING REDUCED OIL LEVELS FOR AGGLOMERATION OF WILLIAMSON COUNTY, ILLINOIS, SEDIMENT

Experiment No.	Recovery Conditions	Overall Oil Conc., %	Recovered Coal Values, %			S_{pyr} Reduction %	g Coal Recovered / g Oil Used
			Recovery[c]	Ash (MF)	S_{pyr} (MAF)		
	Standard agglomeration[a]	10.0	85.6	10.4	2.32		4.17
	with treatment[b]	10.0	83.0	7.3	1.13	51.3	4.52
1	1.0g kerosene/25g sample[b] float once, 0.1g kerosene to residue, float once	4.4	83.0	7.3	0.88	62.1	9.09
2	0.75g kerosene/25g sample[b] float once, 0.1g kerosene added to residue, float once	3.4	89.3	9.5	0.97	58.2	14.3
3	0.75g kerosene/25g sample[b] float once, 0.1g kerosene to residue, float once	3.4	81.3	6.3	0.78	66.4	12.5
4	0.5g kerosene/25g sample float twice, 0.1g kerosene added to residue, float once	2.4	83.2	6.4	0.80	65.5	17.0
5	0.5g kerosene/25g sample[b] float twice, 0.1g kerosene to residue from 2nd float, float once	2.4	78.6	6.1	0.71	69.4	16.5
6	0.5g kerosene/25g sample float six times, 0.1g kerosene to residue from floats 5-6, float twice, 0.2g kerosene to residue 2-4, float once	3.2	75.1	5.8	--	--	11.3
7	0.4g kerosene/25g sample float twice, 0.1g kerosene to residue from 2nd float, float once	2.0	76.9	5.6	--	--	19.2

(a) Using a 10 percent slurry containing 25g of sediment on a dry basis and 10 percent oil concentration based on the sediment weight (i.e., 2.5g).

(b) Sediment slurry was given a 15-minute pretreatment of 0.5 ml 10 percent Na_2S solution/25g sample of sediment (dry basis).

(c) Value based on coal in sediment.

- A series of two air-float separations (the last by air-flotation of the reslurried agglomerated coal recovered initially) as in Experiments 3, 4, 5, and 7 yielded low ash products (5.6 through 6.4 percent ash). A series of six such treatments of the agglomerated coal, each followed by an air-float separation, gave no further ash reduction.
- As the amount of oil used for agglomeration of sediments treated with sodium sulfide was decreased (i.e., 4.4 percent or less compared to 10 percent), the amount of pyrite in the recovered coal decreased. In the absence of sodium sulfide treatment (compare Experiment 4 with results from standard agglomeration) the results are equally dramatic and suggest that only minor additional reductions in pyrites are realized by sodium sulfide treatment. (Compare results from Experiment 5 with those of Experiment 4.) Although no analysis for pyritic sulfur was conducted in the coal recovered in Experiment 7, it too would be expected to be low because the ash value is low.
- Two air-float separations at the same kerosene level (3.4 percent) and pretreatment conditions give lower ash and pyritic sulfur in the recovered coal than a single air-float separation. The apparent coal recovery and coal yield, however, are greater (compare Experiments 2 and 3 where the pyritic sulfur content was reduced from 0.97 to 0.78 percent).

In an attempt to identify an alternative and possibly less expensive source of oil suitable for agglomeration of coal in black water sediments, those originating from non-petroleum sources were given consideration. Cooking oils, preferably waste (spent) cooking oils recovered from fast food chains and others accumulating such material could be one of these items. To determine their behavior during standard agglomeration followed by an air-float separation, the experiments listed in Table 17 were conducted using the Illinois black water. The 10 percent slurry was treated at the 10 and 2 percent oil concentration (partially hydrogenated soybean) based on the dry weight of the sediment charged. The results from those experiments based on coal recovery and yield look encouraging when compared to the results from kerosene. Adjustment of the coal recovery for the weight of oil used (assuming it all stayed with the coal even after drying) reduced the recovery

TABLE 17. YIELD AND RECOVERY OF COAL USING VEGETABLE COOKING OIL FOR THE AGGLOMERATION OF WILLIAMSON COUNTY, ILLINOIS, BLACK WATER

Oil Type	Oil Conc, %	Recovered Coal, %		g coal / g oil
		Recovery[a]	Ash (MF)	
Soybean	10	94.2	6.50	5.3
Soybean	2	83.0	7.35	22.6
Kerosene	2	76.9	5.60	19.2
Kerosene	10	85.6	10.4	4.2

(a) Value based on coal in the feed sediment.

from 94.2 to 78 percent, but at the 2 percent level the recovery remained the same. The behavior of partially oxidized spent cooking oil and the impact of the soybean oil on the pyrite remaining with the recovered coal remain to be investigated.

Treatment of Sediments from McDowell County, West Virginia

Treatment Screening Experiments--

The samples shipped from the coal cleaning plant did not include a sample of the coarse refuse. Therefore no samples of pyrite were available to grind for the treatment screening tests. Some coarse refuse was hand picked from the ROM coal and ground to minus 100 mesh. Examination of the ground material under a binocular microscope did not indicate pyrites present even in the material separated by a "panning" technique. Attempts at mixing a suspension of the ground material in water with kerosene did not yield any material at the kerosene-water interface further suggesting that the available residues contained no pyrites and the screening tests could not be done. However, the black water sediment was assumed to contain pyrites.

It was judged that the treatment sequences tried with the Kentucky and Illinois sediments also would work with the West Virginia sediments.

Sediment Treatment Experiments--

The sequence of treatments outlined for the Kentucky No. 9 sediment was used for the Pocahontas Nos. 3 and 4 sediment using the same procedure as outlined in that section of the report. Typically, sediment samples were treated for 15 minutes after which the pH was adjusted to 8.5 before performing a standard agglomeration followed by separation of the agglomerated coal on a 48 mesh (Tyler) screen. The isolated coal was dispersed and collected by the air-float technique. The coal recovered during agglomeration was extremely powdery to the point of being dusty despite its 30 percent moisture content. This observed behavior was unique to the Pocahontas black water sediment. However, the coal cleaning plant personnel found identical behavior when coal recovery by the "Convertol Process" [4] was tried on the black water.

The results of the pretreatment experiments given in Table 18 suggest that the pyrites in the black water sediment can be reduced by such treatment even at the low levels present in this black water. Treatment at 24 C with sodium

TABLE 18. REDUCTION OF PYRITE IN COAL RECOVERED BY OIL AGGLOMERATION AFTER PRETREATMENT OF BLACK WATER SEDIMENT FROM McDOWELL COUNTY, WEST VIRGINIA

Pretreatment Sequence	Pretreatment Conditions	Recovered Coal Value, %				Percentage Reduction in Pyritic Sulfur	
		Recovery (a)	Ash (MF)	S_{pyr} (MAF)	S_{tot} (MAF)	(b)	(c)
	Black Water Sediment		28.5	0.24	0.83	--	
Number 1	None	76.3	7.56	0.23	0.74	--	(4.2)
Number 2	$NaPO_3 + O_2$, 15 min	65.8	5.97	0.21	0.71	8.7	(21.5)
Number 3	Na_2S only	99.8	5.94	0.21	0.75	8.7	(12.5)
Number 4	$Na_2O_2 + O_2$, 15 min	103.4	8.08	0.20	0.71	13.0	(16.7)
Number 5	$Na_2CO_3 + O_2$, 15 min @ 90 C	96.2	6.57	0.20	0.71	13.0	(16.7)

(a) Values based on coal in feed

(b) Value based on pyrite in recovered coal.

(c) Value based on pyrites in sediments.

sulfide appears to be as effective as the other treatments that used an oxygen sparge at 24 C or at 90 C and probably is simpler to accomplish. Sodium sulfide also appeared to reduce the ash content to 5.94 percent from the 28.5 percent present in black water. (Without reagent the agglomeration lowered the ash to 7.56 percent.) The combined effect produces a desirable recovered coal but there does not appear to be any dramatic reduction in the pyritic sulfur in the recovered coal. This may be because the pyrites are finely disseminated in the ROM coal and are not liberated even in the fine coal present in the black water (88 percent minus 100 mesh). This is strongly supported by the fact that the pyritic sulfur analyses are so uniform over the plus 28 to minus 325 mesh sieve fractions. This fact also helps explain the lack of any pieces of pyrite in the ROM coal sample.

The sulfur and ash values for the coal recovered by agglomeration before and after treatment are compared with ROM and clean coal samples from McDowell County, West Virginia, in Table 19. From these data the following conclusions are made:

- The coal can be cleaned to very low levels of ash and pyritic sulfur that cannot be attained in the coal recovered by standard agglomeration technique.
- The amount of liberated pyritic sulfur in the black water is small and during standard agglomeration it is recovered with the coal.
- The coal recovered after pretreatment or after two air-float collections is lower in ash and pyritic sulfur than the coal recovered by standard agglomeration techniques. It does not appear to be better than the shipped clean coal.

Reduced Oil and Alternative Oil Experiments--

The results from experiments with Kentucky and Illinois sediments in which the amount of oil used for agglomeration was less than 10 percent suggested that such experiments should be tried with the black water sediments from West Virginia. In addition, the impact of the use of soybean oil also had to be examined. The conditions used in the experiments and the results are given in Table 20. The following observations can be made from the data:

- Use of 2.8 to 3.4 percent oil concentrations reduces the pyritic sulfur content in the recovered coal from that obtained during standard agglomeration. Soybean oil appears better than kerosene.

TABLE 19. ANALYSIS OF COAL SAMPLES FROM McDOWELL COUNTY, WEST VIRGINIA, COAL CLEANING PLANT COMPARED TO COAL RECOVERED BY AGGLOMERATION

	Percent		
	Ash (MF)	S_{pyr} (MAF)	S_{tot} (MAF)
Run-of-Mine Coal	26.7	0.28	0.78
Cleaned Coal	6.04	0.10	0.64
Black Water Sediment	28.5	0.24	0.83
Coal Recovered by Agglomeration			
No treatment, screen separation/ single air float	7.56	0.23	0.24
Air float twice	5.63	0.19	0.71
After treatment with Na_2S, screen separation/single air float	5.94	0.21	0.75

TABLE 20. IMPACT ON PYRITIC SULFUR CONTENT OF COAL RECOVERED USING REDUCED OIL LEVELS FOR THE AGGLOMERATION OF PRETREATED AND UNTREATED SEDIMENTS FROM McDOWELL COUNTY, WEST VIRGINIA (Comparison Between Kerosene and Soybean Vegetable Oil)

Experiment No.	Recovery Conditions[a]	Overall Oil Conc, %	Recovered Coal Values, %				S_{pyr} Reduction, %	g Coal Recovered / g Oil Used
			Recovery[d]	Ash (MF)	S_{pyr} (MAF)	S_{tot} (MAF)		
	Standard agglomeration[b]	10	76.3	7.56	0.23	0.74	--	7.84
	with treatment[c]	10	99.8	5.94	0.21	0.75	8.7	7.60
1	0.75g veg oil/25g sample,[c] 0.10g veg oil to residue, Air float recovered coal	3.4	57.1	5.51	0.17	0.66	26.1	12.7
2	0.60g veg oil/25g sample,[c] 0.10g veg oil to residue Air float recovered coal	2.8	52.4	5.31	0.18	0.69	21.7	14.1
3	0.75g veg oil/25g sample 0.10g veg oil to residue Air float recovered coal	3.4	61.0	6.74	0.17	0.67	26.1	13.8
4	0.60g veg oil/25g sample 0.10g veg oil to residue Air float recovered coal	2.8	59.1	7.31	0.17	0.67	26.1	16.3
5	0.60g kerosene/25g sample,[c] Screen 0.10g kerosene to residue Air float Air float recovered coal	2.8	55	5.30	0.19	0.71	17.4	14.9
6	0.60g kerosene/25g sample Screen 0.10g kerosene to residue Air float Air float recovered coal	2.8	54.8	6.79	0.18	0.68	21.7	15.0

(a) Recovery conditions for Experiments 1 through 6 were to recover the agglomerated coal by pouring through screen, perform an air float recovery of coal in the residue after adding 0.1g of oil, and then disperse the combined coal.

(b) The screen recovery of the agglomerated coal was followed by an air float recovery of the separated and redispersed agglomerated coal. Agglomeration used a 10 percent slurry and a 10 percent oil concentration based on sediment dry weight.

(c) Sediment slurry was given a 15-minute pretreatment with 0.5 ml of 10 percent Na_2S solution per 25g of sediment (dry basis) before performing a standard agglomeration.

(d) Value based on coal in feed.

- Treatment with sodium sulfide has little effect on the pyrite level in the recovered coal. However, after treatment the ash values are significantly lowered from those without pretreatment (compare Experiments 1, 2, and 5 with Experiments 3, 4, and 6).
- The yields of coal per gram of oil used are greater at the lower oil concentrations; however, the coal recoveries are lower than when higher oil (kerosene) concentrations are used.
- The coal recovered with the soybean oil did not have any of the undesirable dusty properties of the coal recovered using kerosene as the agglomerating oil.

Treatment of Belmont County, Ohio, Sediments

The effect of pretreatments on the amount of pyritic sulfur in coal recovered from black water sediments from a coal cleaning plant in Belmont County, Ohio, were studied in some detail by Mezey, Min, and Folsom [3]. When the current program began and for about a year after, the only black water sediment available to study was the preserved sediment from Ohio. This material (which is the result of cleaning Pittsburgh No. 8 coal) and the aged and weathered sediments excavated from slurry pond sediments had been well characterized and were well suited for training workers in the art of oil agglomeration.

Treatment of Unweathered Black Water Sediment--

The freshest sediment was used to perform and perfect a wet sieve analysis system and technique used to characterize the sediments to be studied. The wet sieve analysis through minus 325 mesh was performed in replicate samples with the following results:

Isolated Fraction				
Tyler Mesh	Weight Percent Trial 1	Weight Percent Trial 2	Ash, Percent Trial 1	Ash, Percent Trial 2
+28	2.9	2.4	10.5	8.4
28 x 35	1.6	1.6	8.0	8.0
35 x 48	2.5	2.0	8.2	8.1
48 x 65	2.3	2.9	8.5	8.3
65 x 100	3.1	3.0	10.3	8.5
100 x 200	7.8	8.5	11.6	10.0
200 x 325	5.8	6.4	12.0	10.4
-325	74.0	73.2	59.0	59.6
-100 mesh	87.6	88.1		

In the course of this study it became apparent that the ash content of the sieve fractions down to the 200 x 325 mesh size were fairly uniform in their ash content and that only the minus 325 mesh fraction had a large ash fraction, 60 percent. Based on the ash values, 25 percent of the black water sediment was nearly as good in quality as clean coal being shipped. Sufficient minus 325 fraction material was accumulated for study from several wet sieve analyses by settling. (Solids content of the settled solids was about 22 to 24 percent.) Part of the solids was treated by oil agglomeration and air-float separation to yield a coal fraction and coal-free mineral matter fraction. The other part was used to determine the settling rate of the minus 325 material at reduced slurry concentrations. The following agglomeration conditions were investigated, all at 10 percent oil and solids concentration:

1. Sonically dispersed No. 2 fuel oil; high shear mixing followed by two-stage air-float separation of the agglomerated coal
2. Sonically dispersed No. 2 fuel oil, reciprocating shaker followed by two-stage air-float separation
3. High shear mixing dispersed No. 2 fuel oil followed by two-stage air-float separation.

The results of the separation are given below.

	Product Coal		
Experimental Condition	As Percent of Feed	MF Ash, percent	Recovered Coal, percent
1	34.8	5.6	80.7
2	33.8	9.2	75.0
3	32.4	3.1	77.1

These results suggest that although better coal recovery was possible using a dispersed oil, high shear agitation effects better coal recovery and yields a coal with lower ash. Coal recovery is less without the use of predispersed oil (Condition 3) although better mineral matter removal occurs. It must be remembered that the coal recovered originated from minus 325 mesh material with an ash content of about 60 percent. Under Condition 3, the ash content of the coal is the lowest ever recovered from the Ohio black water sediment. These results helped fix the concept of an air-float separation of the recovered coal and its recovery from a second wash dispersion. The coal-free mineral matter suspension was retained for residue studies on improving settling rates and the impact of exposure of the slurry to air on settling behavior.

Treatment of Weathered Sediments--

Experiments were conducted to further characterize weathered, partially dried, slurry pond sediments that had been excavated from an inactive pond in Belmont County, Ohio, near a coal cleaning plant processing Pittsburgh No. 8 coal. The sediment was reslurried with water and allowed to settle to yield a slurry with 60.1 percent solids. The supernatant fluid had a pH of 2.1 and dissolved solids content of 3.5 percent.

The results of two wet sieve analyses given in Table 21 indicate that the material was about 60 percent minus 325 mesh and not unlike the unweathered sediment. The minus 325 mesh solids were collected in about 8.5 liters of water which had a final pH of 2.8 (after about a 140-fold dilution of the occluded water in the sediment. The solid was collected after settling to a slurry with a solids content of 21 percent.

The minus 325 fractions from the two sieve analyses were used to perform three experiments on the recoverability of the coal using a standard oil agglomeration with No. 2 fuel oil followed by two-stage air-float separation:

1. The slurry without any further treatment. The 10 percent slurry had a pH of 3.4 before agglomeration
2. The slurry neutralized to pH 7 to 8 with solid sodium carbonate
3. The slurry washed to a pH near 7.

In the latter case the minus 325 mesh sediment was washed with a volume of water sufficient to give a 10 percent slurry concentration. After the washing period the pH was measured and the slurry filtered and then reslurried in an amount of water equal to the filtrate. After 15 washings the pH remained constant at pH of 6.5. The pH of the slurry was measured at the end of 24-hour periods of stirring, except as noted in Table 22, when it was taken after stirring periods of 2 and 7 days in air. The cause for the reduction of the pH during these periods was not understood, but could be related to the oxidation of the minus 325 mesh size pyrites in the sediment or hydrolysis of iron sulfate already formed.

TABLE 21. WET SIEVE ANALYSIS OF WEATHERED COAL SEDIMENT, BELMONT COUNTY, OHIO

	Trial 1, Percent		Trial 2, Percent	
Sieve Size	Fraction	Cum.	Fraction	Cum.
+30	0.7	0.7	1.1	1.1
30 x 40	3.9	4.6	2.2	3.3
40 x 50	3.5	8.1	3.9	7.2
50 x 70	3.6	11.7	4.4	11.6
70 x 100	7.9	19.6	6.4	18.0
100 x 200	11.5	31.1	11.3	29.3
200 x 325	11.3	42.4	6.8	36.1
-325	57.6	100.0	63.9	100.0

TABLE 22. CHANGE IN pH OF WEATHERED BELMONT COUNTY, OHIO, COAL SEDIMENT SLURRY WITH SEPARATE WATER WASH(a)

No. of Water Washes(b)	Solution/Slurry pH(c)
1	3.1
2	3.7
3	4.2
4	3.2 (2 days)
5	4.7
6	5.3
7	6.1
8	6.7
9	5.4 (2 days)
10	6.6
11	4.4 (7 days)
12	5.1 (7 days)
13	5.4
14	6.2
15	6.5

(a) 25g aged sediment/225g water, original pH = 2.8.

(b) Wash water pH = 6.8.

(c) pH measured after 1-day wash with stirring in air unless otherwise noted.

No coal could be recovered from the unaltered minus 325 mesh sediment by the agglomeration technique at a diluted pH of 3.4. About 28 percent of the feed neutralized with sodium carbonate to pH 7 was recovered as agglomerated coal. About 22 percent of the wash feed was recovered as agglomerated coal. The ash value of the neutralized coal was 7.85 percent while the ash value of the latter was 5.32 percent, suggesting that hydrated iron oxide formed upon neutralization ended up with the agglomerated coal.

RESIDUE CHARACTERIZATION

Any process designed to use oil agglomeration for the recovery of fine coal from coal slurry waste streams or to reduce the environmental threat of slurry pond will be concerned with the disposal of residue from the operation. The composition of the mineral matter suspension and sediments (residues) and their settling rates and retention of oil are all of concern.

Settling Rates

The settling rates of the original coal slurry and of the coal-free residue suspension were obtained by preparing sufficient 10 percent slurry to make up 1700 ml and shaking thoroughly in a 2000 ml graduate cylinder for several minutes. The settling rate was measured by following the level of the clear interface with time. After completion of the settling cycle, the slurry was agglomerated using a 10 percent slurry concentration in a 1-gallon blending vessel. After separation of the agglomerated coal on a 48 mesh screen, the coal was air-floated in about 300 ml of clean water to remove additional mineral matter. The two residue suspensions were combined and contained in a 2000 ml graduate cylinder. The settling rate was measured again.

The settling rates of the black water sediment from Morganfield, Kentucky, and the resulting residue suspension are given in Figure 7. It would appear that the coal-free residue settled at a slightly faster rate than the black water sediment. The addition of 8 ml of 0.1 percent of Superfloc 127® (manufactured by American Cyanamid), a demonstrated clay flocculating agent, did not enhance the settling rate of the coal-free residue suspension. The coal-free sediment reached compression at about 180 minutes whereas the black water

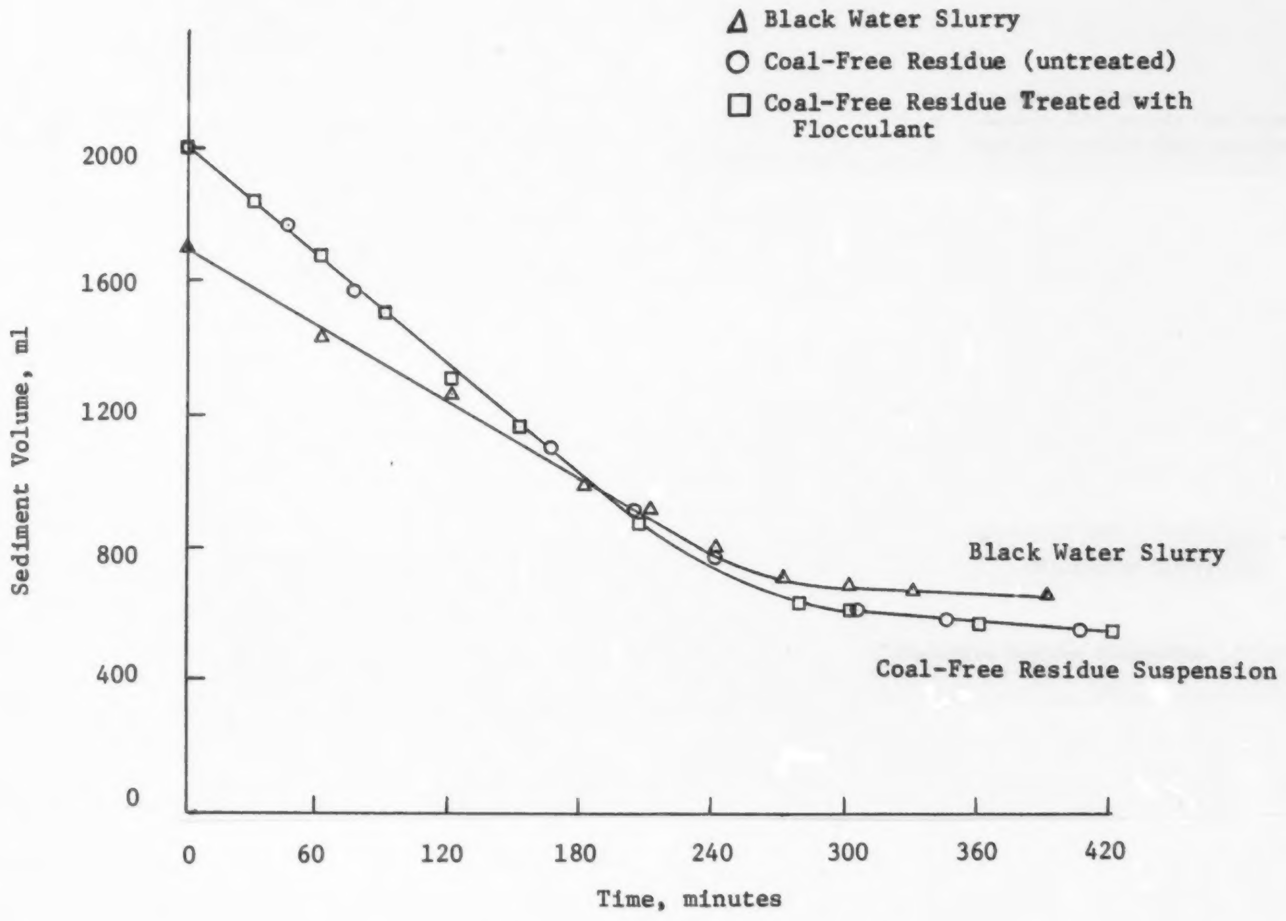


FIGURE 7. COMPARISON OF SETTLING RATES OF A 10 PERCENT SOLIDS BLACK WATER SLURRY FROM MORGANFIELD, KENTUCKY, AND THE COAL-FREE SLURRY AFTER AGGLOMERATION (10 Percent Slurry; 10 Percent Oil Concentration)

sediment took about 200 minutes. Such behavior even with the diluted coal-free sediment is an improvement over the behavior of the untreated sediment.

Differences in the settling rates of the 10 percent solids suspension of black water from Williamson County, Illinois, and coal-free mineral matter suspension are given in Figure 8. The settling rate of the residue after removal of the coal by the standard agglomeration technique appears to be greater than for the black water. They both seem to reach compression at about the same time (100 to 130 minutes).

The settling behavior of a 10 percent solids black water suspension from McDowell, West Virginia, is given in Figure 9 as the average curve of two trials. It would appear that compression begins at about 60 to 70 minutes and is the fastest despite the fine particle size of the black water sediment (i.e., 88 percent minus 100 mesh). The results suggest a faster settling rate for the coal-free slurry remaining after removing the agglomerated coal by means of a screen (initial recovery). When the coal was reslurried in a high shear mixer and recovered by the air-float techniques, a very fine residue remained in suspension. When these solids were combined to yield a suspension volume of 2000 ml (both settled solids were combined and diluted to 2000 ml) the rate of settling was slightly slower but still faster than the black water suspension. The zone of compression appears after about 40 to 50 minutes, also significantly faster than any previous coal-free sediment.

These combined results suggest that improvements in the settling characteristics of the black water might be realized for each of the fresh sediments if the coal values are removed. Agglomeration without pretreatment of the black water sediment would yield a coal-free sediment with less pyrites present. Treatments to reduce the pyrites in the recovered coal would in effect increase their concentration in the coal-free sediments.

Residues from Belmont County, Ohio, were screened to yield minus 325 mesh material for studies as discussed earlier. The impact of the removal of the plus 325 mesh material on the settling rates is illustrated in Figure 10. The removal of the coarse material did reduce the settling rates but gave the impression of faster settling over the reduced range of solids concentrations tried.

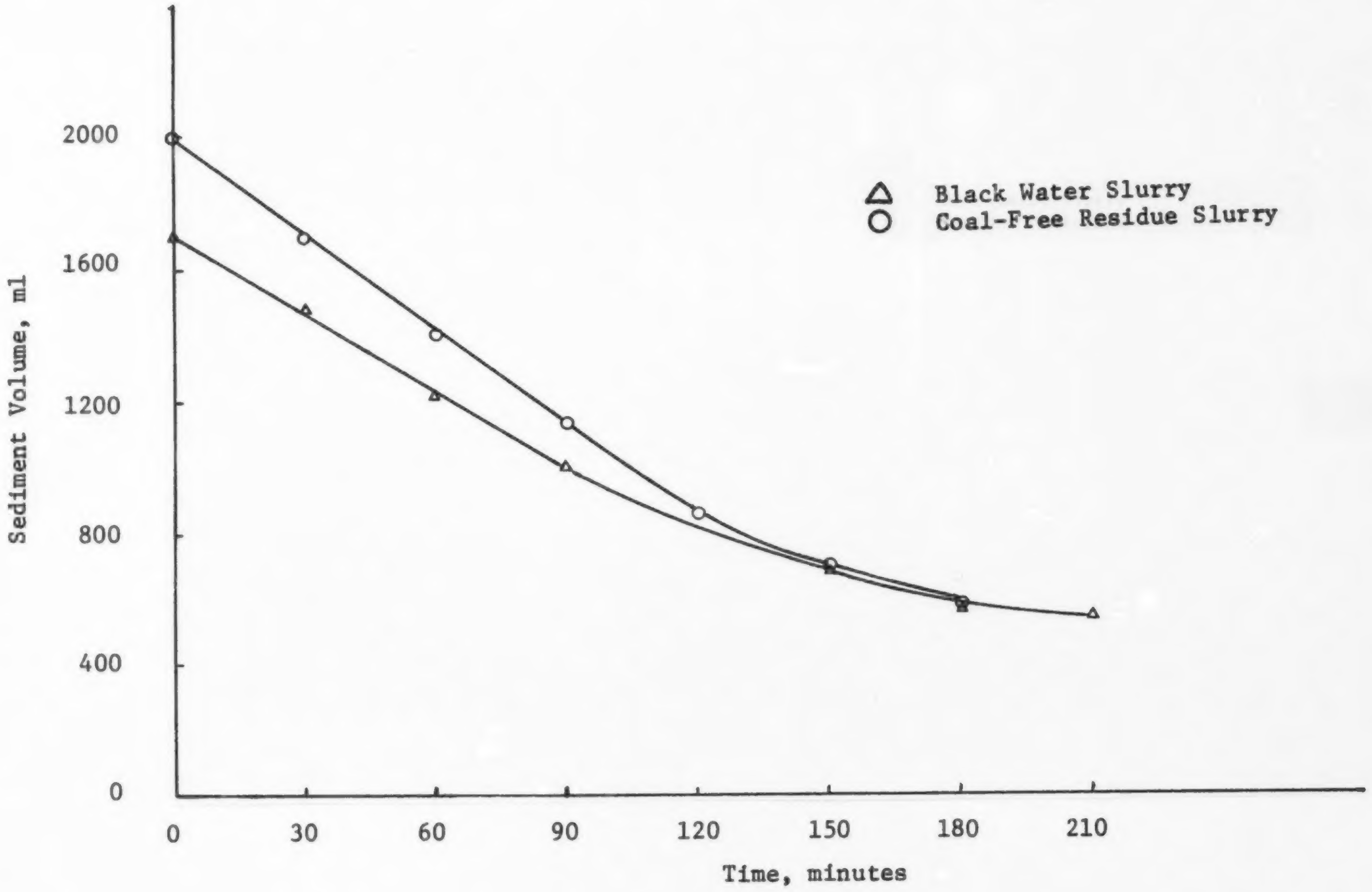


FIGURE 8. COMPARISON OF SETTLING RATES OF A 10 PERCENT SOLIDS BLACK WATER SLURRY FROM WILLIAMSON COUNTY, ILLINOIS, AND THE COAL-FREE SLURRY AFTER AGGLOMERATION (10 Percent Slurry; 10 Percent Kerosene Concentration)

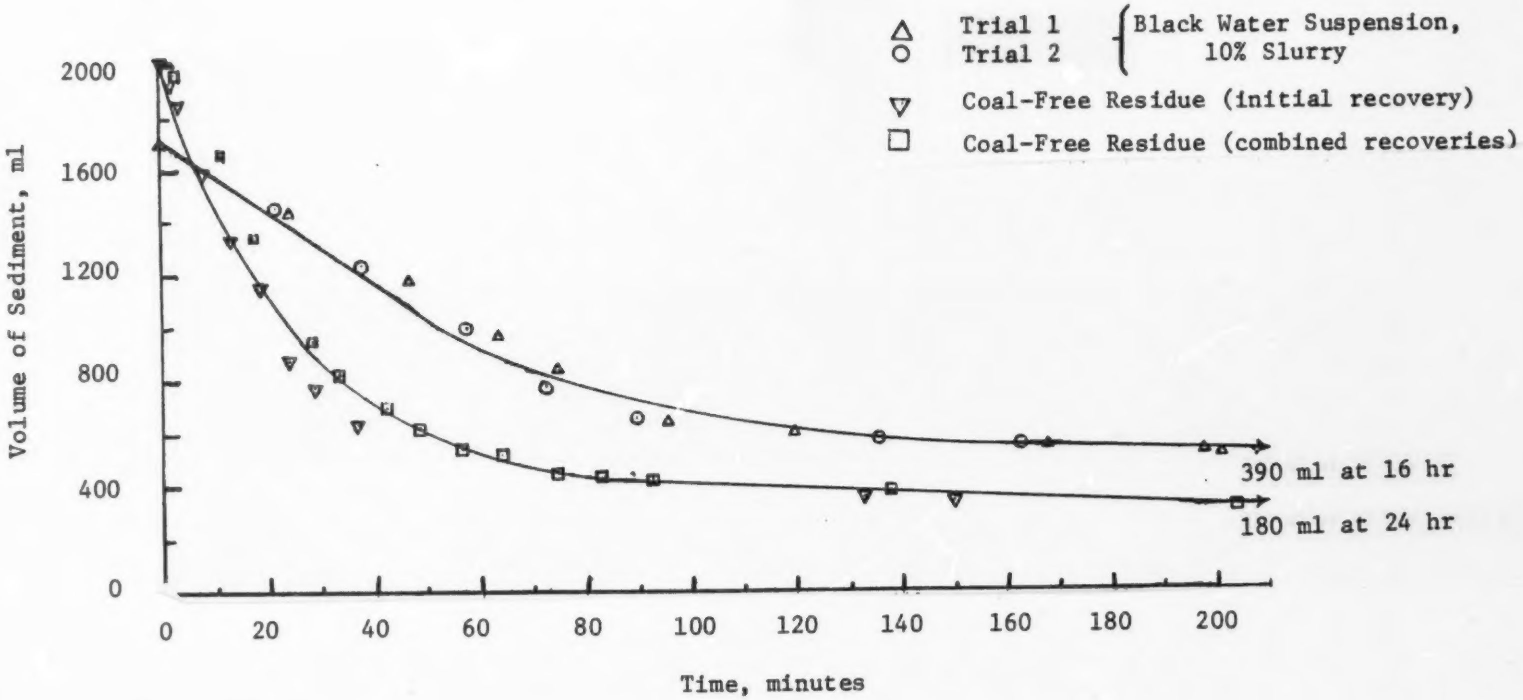


FIGURE 9. SETTLING RATES FOR POCAHONTAS NOS. 3 AND 4 BLACK WATER SEDIMENT AND THE COAL-FREE SLURRY RECOVERED AFTER AGGLOMERATION (10 Percent Slurry; 10 Percent Kerosene Concentration)

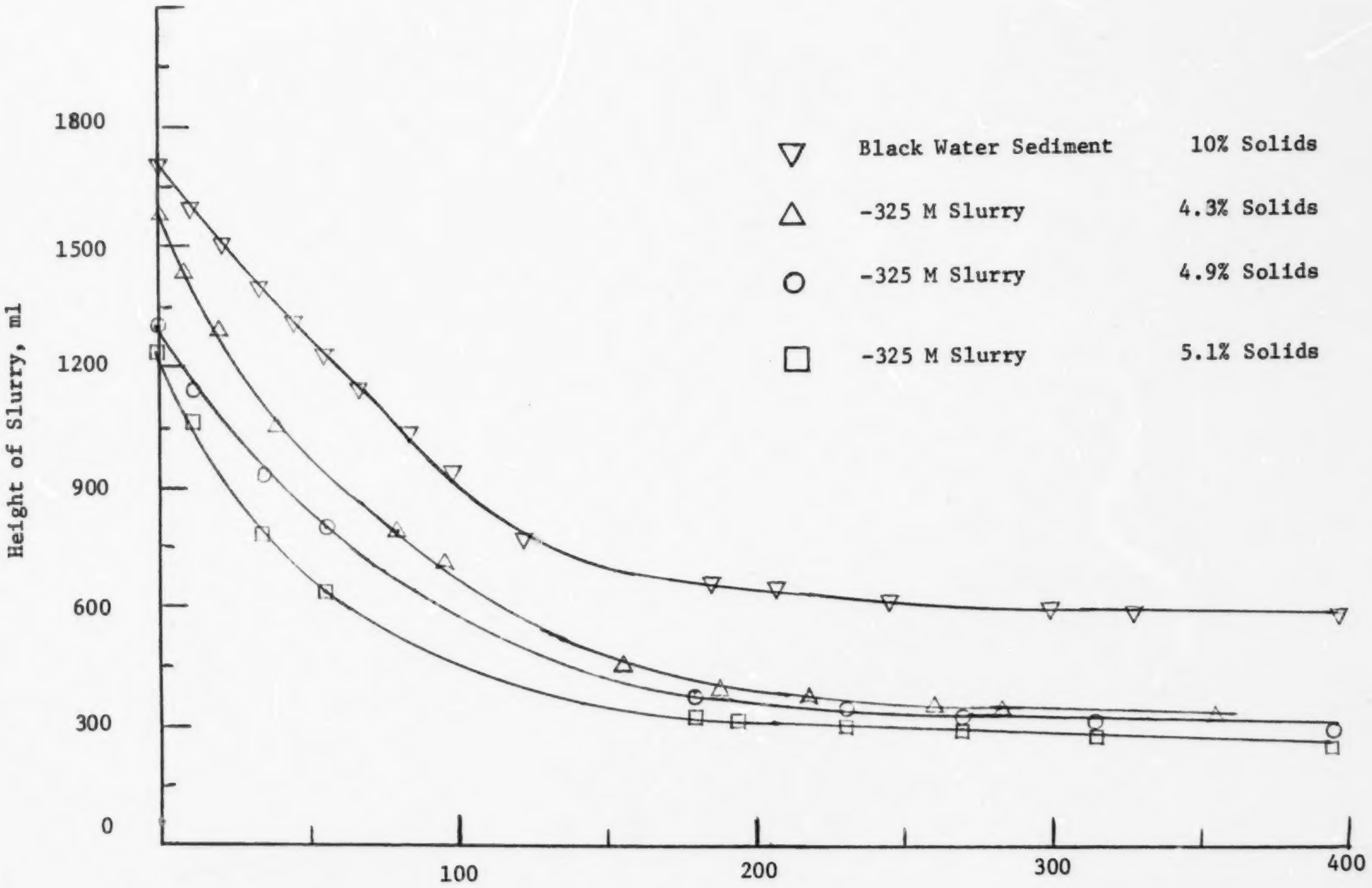


FIGURE 10. SETTLING CURVE OF MINUS 325 MESH SLURRY RECOVERED FROM WET SIEVE ANALYSIS OF BLACK WATER FROM BELMONT COUNTY, OHIO

Chemical Characteristics

In a study on the characterization of black water from eleven coal cleaning operations in the Midwestern and Appalachian region, Aplan, Hogg, and Bradley found the mineral matter associated with the coal to be similar[13]. Each sample contained a large amount of "illitic" material, with most samples containing lesser amounts of kaolinite, chlorite, calcite, quartz, and pyrite. "Illitic" minerals are illite, montmorillonite, and interstratified illite montmorillonite in varying ratios.

Reaction with Air--

Because it was demonstrated that the minus 325 mesh fraction of the black water sediment from Belmont County, Ohio, was about 60 percent ash, it was believed that the impact of the presence or absence of coal on the air oxidation and subsequent settling rate might be determined by comparing the coal-free sediment behavior to that of the entire black water sediment. The results of the aeration of two minus 325 mesh slurries--one with coal present and the other with the coal removed by agglomeration--for a period of 50 days is given in Table 23. About 250 ml of a 10 percent slurry were sparged using 30 ml/min of air while being stirred continuously. The pH was measured periodically and water lost was replenished. The data in Table 23 suggest that the pH of the coal-free sediment slurry did not decrease after 42 days but that the pH of the coal-containing slurry did. Such behavior suggests that the source or the mechanism of acid formation is altered by removing the coal i.e. removal of the finely divided pyrite and coal. (This assumes that pyrite oxidation was the source of the acid and reduction of pH.) The reason the pH did not change through the 28th day was probably due to the buffering capacity of the clay minerals.

Several attempts at performing sedimentation tests with the aerated slurries were made. The measured settling rates were very irratic and were not considered to be valid because some solids were lost during the aeration periods. Since the point of zero charge (zeta potential) for silicate minerals such as clays is usually below a pH 4, the settling rate of the slurries at pH < 4 would be expected to be faster in acidic media. However, this was not considered to be universally applicable to all illitic clays [13].

TABLE 23. CHANGE IN pH OF SLURRIES OF MINUS 325 MESH COAL-FREE AND COAL-CONTAINING SEDIMENT DURING AERATION

Aeration Period, days	Minus 325 Mesh Coal Sediment Fraction		Dissolved Solids Conc, mg/ml in	
	Coal Free[a] (Sample A)	Coal Present (Sample B)	(A)	(B)
0	7.90	7.90		
1	7.60	7.35	0.11	0.11
4	7.35	6.95		
5	7.40	6.90		
7	7.35	7.00	0.45	0.66
8	7.30[b]	7.20[b]		
10	7.10	6.90		
11	7.30	7.00		
12	6.80	6.70		
13	6.90	6.65		
	7.10[b]	6.85[b]	2.11	2.56
14	7.10	6.85		
20	7.15[b]	7.05[b]		
28	7.30	6.70		
42	6.30	4.35		
50	7.40[c]	4.60[c]		

(a) Coal removed by oil agglomeration.

(b) pH of clear supernatant after dilution to original volume with water with a pH = 6.8.

(c) pH after dilution with water with a pH = 7.6.

Coal cleaning plant refuse from the cleaning of low sulfur coal from the Appalachian region has been characterized to have less than 1 percent pyrite or marcasite by Wewerka, et al. [14]. Clay minerals and quartz represent 50 weight percent of the refuse composition. The combination of low pyrite with calcite present suggests that the refuse has a lower acid-generating potential upon weathering than say a refuse with high sulfur content with similar mineral composition, e.g., refuse from the Illinois basin but without calcite present.

Oil in the Residues--

How much oil stayed with the sediments (residues) remaining after agglomeration was not determined. The fate of oils, such as No. 2 fuel, in water was studied by Zürcher and Thüer [15] in 1978. They found that certain aliphatic hydrocarbons with molecular weights above 250 were adsorbed onto kaolinite, and that oil droplets on settable kaolinite particles were agglomerated with suspended minerals to concentrations exceeding threefold the values of the adsorptive loading of oil on kaolinite. They concluded that the fate of oil in water is strongly directed by fast dissolution, adsorption, dispersion, agglomeration and weathering processes which are followed by sedimentation and biochemical alteration of an oil phase. It is anticipated that the fate of the oils used in agglomeration would be similar, except that the major concentration of oil would end up with the coal and the remainder with the sediment.

SECTION 6

ENGINEERING ANALYSIS OF OIL-AGGLOMERATION TECHNIQUES APPLIED TO BLACK WATER EFFLUENT CONTROL

INTRODUCTION

The implementation of any new industrial process that has been demonstrated to be technically effective will depend to a high degree on factors that relate to the practical and economic feasibilities of its integration into existing processing operations. This axiom is certainly relevant to oil-agglomeration techniques applied to fine coal recovery as a method of effluent control. Although laboratory results indicate that oil agglomeration combined with air flotation is highly effective in achieving a good partitioning between the coal product and minerals fraction, these techniques need to be designed into waste-coal processing schemes that are likely to perform well at the industrial scale and that are economical to construct and operate.

The main goals of this section are to discuss possible unit processes that could be effectively combined with oil agglomeration to achieve good coal separations, to present a conceptual design of a process scheme requiring relatively low energy inputs, and to perform a preliminary economic analysis on the selected system.

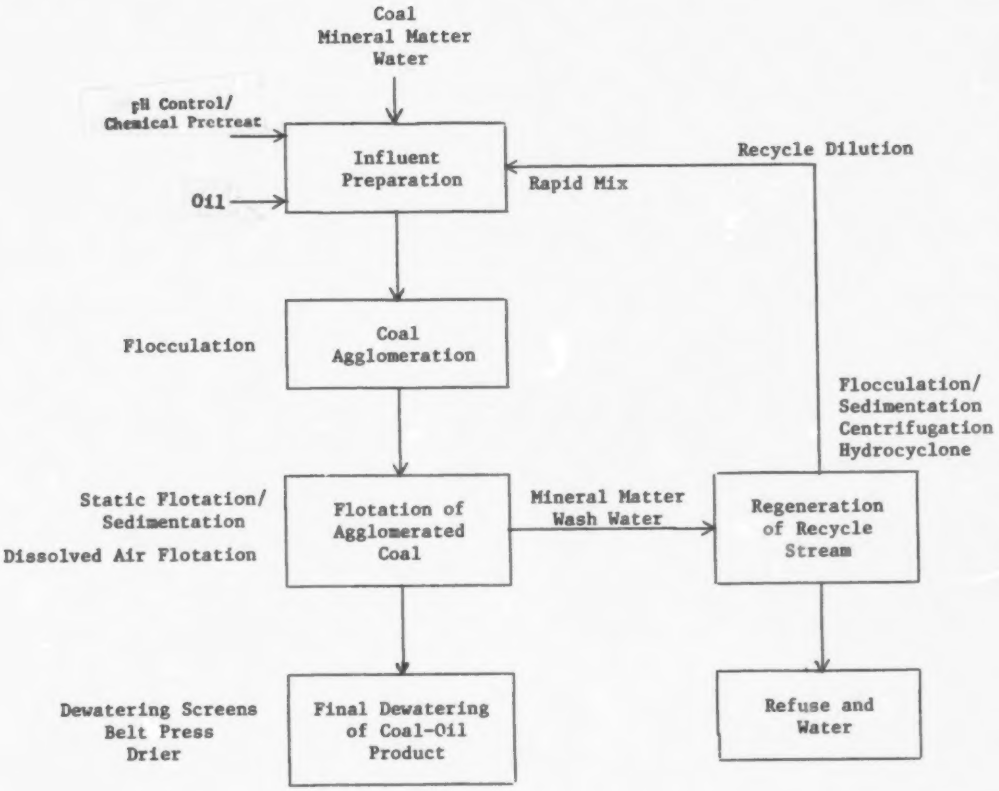
PROCESSING OBJECTIVES

As previously discussed, fine coal-mineral sediments wasted from coal preparation operations are likely to be discharged from a final gravity thickener at a slurry concentration of 30 to 35 percent total solids (TS). Because laboratory results of this study indicate that the oil-agglomeration technique works well on 10 percent TS coal slurries, a dilution of more than 3:1 of coal slurry influents would undoubtedly be required. Compositional characteristics of coal-waste streams may vary, but the coal fraction usually comprises about half of the total suspended solids. The pH of this stream may vary between 6.5 and 8.5. In light of these and other waste coal stream characteristics previously presented in this report, a number of general treatment objectives can be clearly identified which should serve as the basis for the selection and design of coal agglomeration/separation process schemes. These objectives are as follows:

- The preparation of the influent stream through the complete mixing of the influent coal waste stream with the correct amounts of dilution water, base and chemical pretreatment followed by oil addition to achieve the desired solids consistency (10 percent TS), pH (7.0-7.5), pyrite treatment, and oil-solids contact
- The effective flocculation of agglomerated coal-oil particles
- The efficient flotation and separation of the agglomerated coal from the bulk solution
- The partitioning of agglomerated coal from the discrete mineral particle fraction including the pyrites
- The removal of suspended solids from the coal wash water with the net result of providing an acceptable-quality recycle stream for influent dilution
- The thickening of mineral particulate sludges for ease of solids handling
- The production of a dewatered, high-quality, agglomerated coal product that is suitable for pelletizing and use as a high-Btu fuel.

PROCESS DESCRIPTION

In achieving the above processing objectives, the coal-oil-agglomeration process must be used in combination with a number of unit processes that are compatible with its operation. A general flow diagram outlining the basic operations that would be required for the oil-agglomeration treatment of concentrated coal-waste streams is shown in Figure 11. It is significant that no specialized equipment need be required; each process step shown in the diagram could be performed with equipment items that are commercially available to the coal industry. In particular, various physical separation processes have been used extensively in the treatment of coal waste streams in the past; these



Coal
Mineral Matter
Water
pH Control/
Chemical Pretreat
Oil
Influent Preparation
Rapid Mix
Recycle Dilution
Flocculation
Coal Agglomeration
Flocculation/
Sedimentation
Centrifugation
Hydrocyclone
Static Flotation/
Sedimentation
Dissolved Air Flotation
Flotation of Agglomerated Coal
Mineral Matter
Wash Water
Regeneration of Recycle Stream
Dewatering Screens
Belt Press
Drier
Final Dewatering of Coal-Oil Product
Refuse and Water

FIGURE 11. GENERAL TREATMENT STEPS REQUIRED FOR AGGLOMERATED COAL SEPARATION FROM COAL PROCESSING WASTE STREAMS

have included sedimentation, dissolved air flotation, dewatering screens, centrifugation, hydrocycloning, and belt press methods. The applications of these unit processes to coal preparation are discussed in considerable detail in the literature [16-19]. Because waste coal sediments usually contain fine particulates of the sizes and characteristics of clay particles, a fraction of these fines is not easily removed from waste streams with sedimentation alone. Hence, flocculants, such as aluminum sulfate, are often used by the coal industry to achieve higher efficiencies of suspended solids removal.

As indicated in Figure 11, the conventional unit processes described above could be used with oil agglomeration to comprise an effective waste-coal recovery scheme. In the first step of the coal-processing train, the influent coal stream at 35 percent TS is mixed with a base for pH control, chemical for pyrite treatment, oil and recycled water (for dilution) in a rapid mix tank for 30 seconds, resulting in a 10 percent TS slurry. This high-shear mixing step, employing shear velocities of 750 to 1000 fps/ft, ensures good contact between coal particles and the oil. The suspension is then transferred to a flocculation tank where it is mixed for about 10 minutes at lower speeds to encourage particle-particle collisions and coagulation of the coal. The agglomerated mixture is then transferred to static flotation basins or to a dissolved air flotation system to separate the floated agglomerated coal from the mineral tailings. The oil-coal product is then skimmed off the surface of the bulk solution and subjected to dewatering prior to pelletizing. The inevitable by-product of this kind of waste coal processing is the generation of large volumes of dilution water turbid with the clay and sediment separated from the coal. The suspended solids of this fraction could be removed by employing centrifugation, hydrocycloning, or flocculation and sedimentation techniques with the result of upgrading an effluent suitable for use as recycled dilution water for the waste-coal influent [16,20-23].

PROCESS SELECTION

As one might imagine, many combinations of solids separation unit processes may be used with oil agglomeration to achieve coal separation. In the selection of unit processes, however, the projected capital expenditures, operating and maintenance costs, chemical requirements, and anticipated energy inputs must all be considered, as well as the flow scheme's compatibility with the oil-agglomeration process itself.

The most critical steps in agglomerated coal separation schemes are undoubtedly the methods employed for coal flotation and washing methods. From laboratory observations it is estimated that up to 10 percent of the mineral matter of an agglomerated slurry introduced into a flotation unit could be entrained in oil-coal flocs. To ensure a high quality oil-coal product of a low mineral content, it was considered necessary to employ a two-staged coal washing scheme utilizing the flotation properties of oil-agglomerated coal. The flotation recovery of agglomerated coal can be achieved with either static flotation/sedimentation tanks or with the commercially available dissolved air flotation (DAF) system [17,23].

In the DAF system, air is dissolved in a portion of the effluent water stream under pressure of several atmospheres and is combined with the influent stream at atmospheric pressure. The drop in pressure to atmospheric levels and the concomitant reduction in air solubility causes the emergence of small bubbles which attach to particulate matter and promote flotation [23]. Such a system would be highly effective in the partitioning of agglomerated coal, as reflected in the laboratory results presented in this report. Agglomerated coal recovery efficiencies would be expected to be significantly greater with the DAF system as compared with those efficiencies attainable with static separation basins.

The two-stage agglomerated coal washing scheme which appeared most attractive for implementation consisted of a static flotation/sedimentation tank used for a rough separation of mineral matter from the coal followed by a resuspension of the skimmed, agglomerated coal in more dilution water to form a slurry of 10 percent TS which would be transferred to a DAF system for a final "polishing" separation of the coal product from entrained mineral matter. Although the DAF system would be highly effective in providing good flotation separations of the agglomerated coal with superior oil-coal recovery efficiencies, only one of these units was used because of the high capital cost and energy inputs associated with this type of unit. On the other hand, the use of a static flotation/sedimentation unit as the initial coal flotation separation step would reduce the capital costs of the scheme, though coal recovery efficiencies would be slightly reduced. Such cost relationships are presented in the economics discussions of this section.

Skimmings of the final coal product from the DAF system would require some dewatering to eliminate excess dragout water. Because the oil-coal product would be hydrophobic in nature, dewatering the coal from 20 percent to a 10 percent moisture content may be accomplished with a 50-mesh dewatering screen.

Finally, conventional flocculation with alum followed by sedimentation were the processes chosen for the removal of suspended solids from the underflow streams of the flotation units to provide the recycle dilution water used by the process scheme. This water treatment technique was chosen because of its proven effectiveness in removing fine suspended solids in the coal industry [16] and because the energy required for its operation is generally less than half of the electricity demanded of hydrocyclones and centrifuges. The sludges generated from the sedimentation tank (consisting of alum, mineral matter, and small amounts of unrecovered coal) could be deposited in existing refuse holding ponds or pumped back into mining areas as fill material.

On the basis of the above considerations and on the basis of the laboratory results presented in this report, a coal agglomeration processing scheme was developed as shown in Figure 12. Typical coal preparation operations generate fine waste coal at rates exceeding 50 TPH; the waste coal is usually discharged as a thickened sludge at a consistency of 30 to 36 percent TS. The coal recovery scheme of Figure 12 is sized for an input of 50 TPH of coal and mineral matter, received from the coal plant thickener as a 34.5 percent TS slurry. In this example it is assumed that the coal fraction comprises about 50 percent of the total particulate solids. Coal particles to be recovered will contain 4 to 6 percent of noncombustible components; however, the inherent ash content of the coal particle matrix is not considered a part of the mineral fraction in this discussion. Rather, the mineral fraction referred to here is comprised of discrete mineral particulates, including clays and sediment, formed in waste coal sludges.

Mass flows throughout the entire coal cleaning scheme are indicated in the diagram and are based upon a number of assumptions:

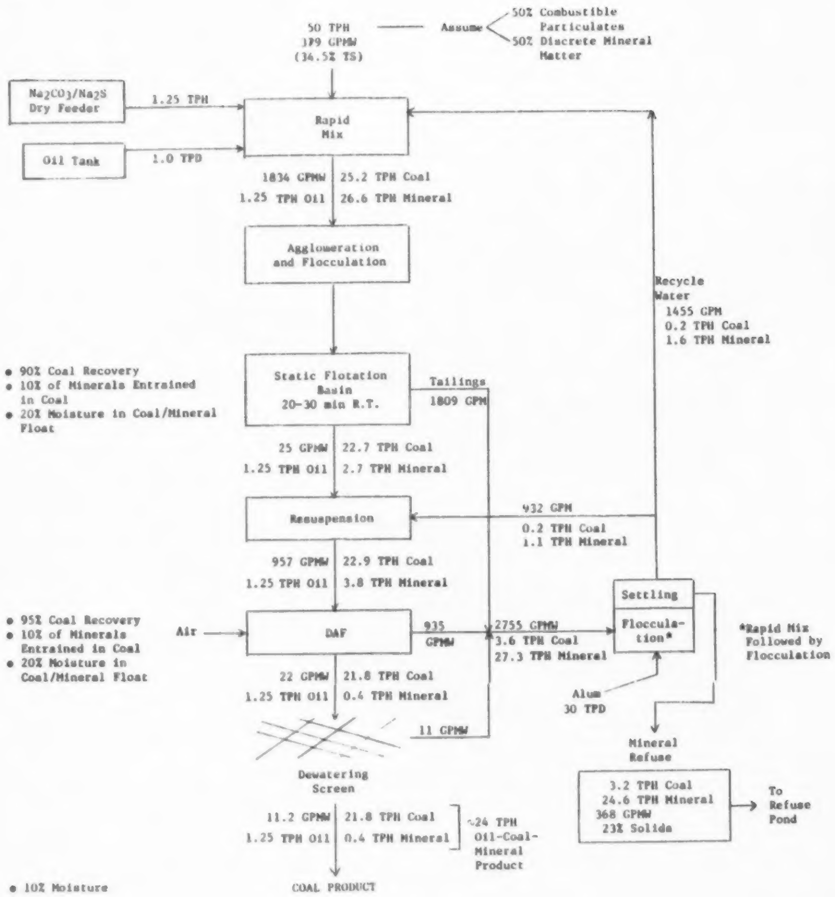

50 TPH
379 GPMW
(34.5% TS)
Assume
50% Combustible Particulates
50% Discrete Mineral Matter
Na$_2$CO$_3$/Na$_2$S Dry Feeder
1.25 TPH
Oil Tank
1.0 TPD
Rapid Mix
1834 GPMW
1.25 TPH Oil
25.2 TPH Coal
26.6 TPH Mineral
Agglomeration and Flocculation
Recycle Water
1455 GPM
0.2 TPH Coal
1.6 TPH Mineral
• 90% Coal Recovery
• 10% of Minerals Entrained in Coal
• 20% Moisture in Coal/Mineral Float
Static Flotation Basin
20-30 min R.T.
Tailings
1809 GPM
25 GPMW
1.25 TPH Oil
22.7 TPH Coal
2.7 TPH Mineral
Resuspension
932 GPM
0.2 TPH Coal
1.1 TPH Mineral
957 GPMW
1.25 TPH Oil
22.9 TPH Coal
3.8 TPH Mineral
• 95% Coal Recovery
• 10% of Minerals Entrained in Coal
• 20% Moisture in Coal/Mineral Float
Air
DAF
935 GPMW
2755 GPMW
3.6 TPH Coal
27.3 TPH Mineral
Settling
Floccula-tion*
*Rapid Mix Followed by Flocculation
Alum
30 TPD
22 GPMW
1.25 TPH Oil
21.8 TPH Coal
0.4 TPH Mineral
11 GPMW
Dewatering Screen
Mineral Refuse
3.2 TPH Coal
24.6 TPH Mineral
368 GPMW
23% Solids
To Refuse Pond
11.2 GPMW
1.25 TPH Oil
21.8 TPH Coal
0.4 TPH Mineral
~24 TPH Oil-Coal-Mineral Product
• 10% Moisture
COAL PRODUCT

FIGURE 12. CONCEPTUAL FLOW SCHEME OF A 50 TPH OIL-AGGLOMERATION WASTE COAL RECOVERY FACILITY

- Suspended solids concentrations of coal/mineral matter slurries prepared in the influent rapid mix tank and in the resuspension rapid mix tank are prepared as 10 percent TS solutions.
- About 100 ppm of soda ash/sodium sulfide is added to this influent rapid mix system for the pH control/chemical pretreatment necessary to enhance pyrite wetting and to enhance alum flocculation and sedimentation in the treatment of turbid recycle water.
- Oil introduced into the system (0.024 tons per ton solids processed) is rapidly attached to coal particles and is conserved through the system as a component of the recovered coal float.
- Coal recoveries are assumed to be 90 percent in the static flotation basin and 95 percent in the dissolved-air flotation (DAF) unit.
- About 10 percent of discrete mineral matter introduced into the static flotation basin and the DAF system is entrained in the coal float.
- The moisture of agglomerated coal skimmed from the static and dissolved-air flotation basins is 20 percent of the total weight.
- About 90 percent of the discrete mineral and coal particulate matter in the recycle stream can be removed with flocculation and sedimentation.
- Mineral and alum sludges generated by the process scheme can be deposited in existing storage pond facilities.

Using the processing scheme of Figure 12, it is expected that good coal demineralization and coal recovery can be achieved. In the flow scheme, a waste coal sludge containing 50 percent mineral matter (mostly clay and sediment) is converted to a quality fuel containing less than 2 percent of discrete, mineral impurities (not including the ash content of the coal which approximates 5 percent by weight). The total noncombustible fraction of the recovered coal product should remain at levels below 7 or 8 percent. Furthermore, of the 25 TPH coal introduced to the system, about 21.8 TPH of a high quality coal product can be obtained, a yield greater than 87 percent. It is expected that about 1.25 TPH of No. 2 fuel oil, a mixture of No. 1 and No. 6 fuel oils, or a vegetable oil should be introduced into the influent rapid mix tank and that virtually all of the oil can be recovered in the agglomerated coal product. The total amount of the oil-coal fuel produced would exceed 24 TPH.

In the course of coal refinement, the process scheme generates considerable amounts of wastewater, almost 3,300 gallons per ton solids processed (14 tons wastewater per ton solids input). About 90 percent of the mineral and unrecovered coal particulate matter is removed in the recycle water treatment scheme consisting of flocculation with aluminum sulfate (pH 7.0-7.5) and sedimentation. Suspended solids removal efficiencies of 85 to 95 percent are commonly reported in the literature for sedimentation processes applied to coal waste water treatment [16]. Clarifier underflow solids concentrations of greater than 30 percent are also reported for sedimentation systems utilizing alum flocculation [16]. Water balances on the system can be maintained by discharging a 23 percent TS sludge from the refuse clarifier. Therefore, additional sludge thickening, via centrifugation or hydrocyclone, is not required.

DESIGN CONSIDERATIONS

As previously mentioned, most of the processes included in the coal agglomeration processing scheme of Figure 12 are simple in nature, employing conventional water-treatment and sludge-handling methods. A list of the major pieces of equipment used in the processing scheme is presented in Table 24. For each of the unit processes a conceptual design was developed which includes equipment sizing, calculations of important dimensions based upon design criteria, specification of major components and materials, and the identification of possible equipment suppliers [20,21,23,25]. This information is summarized in Table 24 for a 50 TPH coal agglomeration plant. All of the loadings and assumptions specified for each of the unit processes of the coal-agglomeration scheme were incorporated in the conceptual design.

Most of the processes are based upon design criteria similar to guidelines used in the design of municipal water treatment plants. Some departures, however, were warranted. First, the oil-agglomeration unit consisted of a flocculation tank with a 10-minute retention time, much shorter than conventional flocculation times of 30 minutes or more. From laboratory observations, oil agglomeration of coal particles would be expected to proceed at a much faster rate than, say, the alum agglomeration of mineral matter. Second, the retention time of the sedimentation tank used as a static flotation basin

TABLE 24. MAJOR EQUIPMENT LIST FOR THE 50 TPH OIL-AGGLOMERATION COAL RECOVERY PROCESS SCHEME

Item	No. of Units	Unit Loading	Unit Capacity	System Dimensions	Additional Design Information	Possible Equipment Suppliers
I. Rapid Mix	1	3.0 MGD	162 ft^3 (liquid)	L x W x D 5' x 4' x 10'	• Velocity gradient, G - 700 fps/ft • Axial impeller flash mixer used, 4 hp, 100 rpm • x 35 second retention time (RT) • Concrete basin	• Linkbelt • Dorr-Oliver • Walker Process
II. Chemical Addition						
• Oil	3		144,000 gal	24' x 32' dia. Cone roof storage tank	• Assume 0.024 tons/ton coal solids processed • 30 day supply • Hot rolled steel storage tank • Two metering pumps at 400 GPH will be used, variable speed	• BIF (pumps) • Calgon • Envirotech
• Soda Ash/Na_2S	1		1 TPD		• Assume 100 ppm dose is required for pH control to pH 7.0-7.5 • Bag elevator • Slider conveyor • Bag ripper • Chemical feeder • Metering • About 300 ft^2 storage area needed for a 20 day supply	• Wallace & Tiernan • BIF • Dresser
III. Oil Agglomeration	2	1.5 MGD	1,600 ft^3	12' x 13' dia.	• R.T. = 10 min • G = 60 fps/ft • 4 paddles, 6" wide, 8 ft long on one vertical shaft • Mechanical mixer, 0.3 hp • Concrete basins	• Ecodyne Corp. • FMC
IV. Static Flotation/ Sedimentation	1	3.0 MGD	10,000 ft^3	50' x 20' x 10'	• R.T. = 30 min • Sedimentation basin has a 12:1 slope • Mechanical skimmer: chain with flight collector • Sludge scrapers included • Concrete basin	• Dorr Oliver • Walker Process • FMC

TABLE 24. (Continued)

Item	No. of Units	Unit Loading	Unit Capacity	Systems Dimensions	Additional Design Information	Possible Equipment Suppliers
V. Resuspension Tank	1	1.5 MGD	69 ft^3	4' x 4' x 6'	• G = 700 fps/ft • R.T. = 30 seconds • Axial impeller flash mixer, 2.0 hp • Concrete basin	• Linkbelt • Dorr-Oliver • Walker Process
VI. DAF	1	1.5 MGD	Surface area A_s = 430 ft^2	29' x 15' x 10'	• Surface loading L = 3500 gpd/ft^2 • Float R.T. = 30 min	• Komline-Sanderson
VII. Dewatering Screen	1	12 TPH			• Three units operated in parallel	• Walker Process • FMC • Ecodyne
Recycle Water Treatment						
VIII. Rapid Mix	1	4.1 MGD		5' x 5' x 7.7'	• G = 700 fps/ft • R.T. = 30 seconds • Axial impeller flash mixer used, 5.0 hp • Concrete basin	• Linkbelt • Dorr Oliver • Walker Process
IX. Alum Addition	1		3.0 TPD		• Assume a 100 ppm alum dose is needed for flocculation • Bag elevator • Slider conveyor • Bag ripper • Chemical feeder • Metering • About 900 ft^2 storage area is needed for a 20-day supply	• Wallace Tiernan • BIF • Dresser
X. Flocculation	1	4.1 MGD	11,520 ft^3	[illegible]4' x 24' x 13'	• R.T. = 30 min • Tapered flocculation - Influent tank section G = 60 fps/ft - Middle tank section G = 40 fps/ft - Effluent tank section G = 20 fps/ft • Paddle areas - Influent section: 36 ft^2 - Middle section: 20 ft^2 - Effluent section: 10 ft^2	• Ecodyne Corp. • FMC

TABLE 24. (Continued)

Item	No. of Units	Unit Loading	Unit Capacity	Systems Dimensions	Additional Design Information	Possible Equipment Suppliers
XI. Sedimentation	1	4.1 MGD	68,580 ft^3	Diameter: 100' Side depth: 11' Center depth: 15' Freeboard: 2'	• R.T. = 3.0 hr • Overflow rate = R; $R \simeq 0.37$ gpm/ft^2 • Velocity through tank = v; v = 0.5 ft/min • Weir overflow rate: 0.013 MGD/ft • Mechanical sludge removal equipment • Concrete basin	• Dorr Oliver • Walker Process • FMC
XII. Pumps						
• Centrifugal	2		4000 GPM		• Large pumps for recycle water transfer	• Goulds Pumps • BIF
• Diaphragm	2		400 GPM		• Mineral sludge transfer to holding pond	• FMC
• Low capacity	1		30 GPM		• Water transport from dewatering screens	• Goulds Pumps • BIF • FMC
XIII. Coal Product Handling & Storage Facilities						
• Storage Area 1			24 TPH	Storage area: 9700 ft^2 Shelter: 70' x 140'	• Storage area consists of a pole building positioned over a concrete slab which is drained into a wet well outfitted with a sump pump to control excess oil/wastewater drainage. Excess oil may be reused for agglomeration • A 10 GPM pump is included	
• Coal Conveyors	1		24 TPH	Total Length: 100'		• Dresser Industries • FMC • Keene Corp.
• Front End Loaders (Bobcats)	2				• Used for moving oil-coal solids within the storage facility	

(0.5 hours) need not be as long as the retention times used for conventional settling (> 4 hours) because oil-coal flotation, the principal objective of this unit process, would be expected to proceed at a rapid rate. Lastly, the coal processing literature seems to indicate that clarifier overflow rates as high as 0.38 gpm/ft^2 and hydraulic retention times as low as 1.5 hours are tolerable for the sedimentation of alum-flocculated coal processing particulates [16]. The alum sludge clarifier employed in the recycle water treatment scheme was designed at an overflow rate of 0.38 gpm/ft^2 and a retention time of 3.0 hours. Municipal water treatment sedimentation systems are usually designed at overflow rates of 0.25 to 0.37 gpm/ft^2 and hydraulic retention times in excess of 4.0 hours, probably because of the higher treatment demands placed on drinking water supplies [20,21]. The theoretical aspects of high-performance flocculation and sedimentation of waste stream particulates are discussed in depth in the literature [22,24].

The major loadings on the system include about 3.0 MGD of coal waste influent and dilution water introduced into the initial coal agglomeration/separation stage, about 1.5 MGD of coal skimmings and more recycled dilution water transferred to the DAF coal separation stage, and about 4.1 MGD of recycle water treated by alum flocculation and sedimentation.

Chemical feed rates were based upon design flows and certain assumed dosages. Dosages used for the coal agglomeration scheme included 0.024 tons of oil per ton of coal waste solids introduced, 100 ppm of soda ash added to the combined influent-recycle stream-oil agglomerated mixture, and 100 ppm of alum required for flocculation and sedimentation of suspended solids in the entire recycle stream. These chemical dosages would, of course, be subject to change from one type of coal sludge to another. Actual laboratory tests to determine buffering requirements, oil-agglomeration characteristics, and alum-flocculation and sedimentation behavior of wash water particulates would be needed to establish effective chemical dosages.

ECONOMIC ANALYSIS

The economics of coal agglomeration and recovery should depend on a number of factors that would be difficult to generalize for all operations. Because significant differences can exist among coal preparation process schemes and the waste coal sludges generated, the costs of integrating coal agglomeration process trains into coal plants may vary. However, the most important aspects of oil-agglomeration economics related to waste coal recovery can be addressed and costs can be based on assumptions that are realistic for a wide cross section of the industry.

The costing approach taken here was to consider the oil-agglomeration scheme of Figure 12 to be an add-on to an existing coal preparation facility in which waste coal sludges were previously thickened and transferred to a refuse pond. Thus, the oil-agglomeration process scheme would be placed between the final plant thickener (producing sludges at 34.5 percent TS) and the refuse pond. Such a location for the coal recovery system would have the advantage of allowing existing coal sludge thickening facilities to be used to reduce volumes of waste sludges to be handled in the coal recovery process, thereby reducing basin size requirements for the more expensive dissolved air flotation units, as well as reducing capital cost and chemical dosages. It is further assumed in this economic analysis that the existing refuse pond would be available to receive wasted mineral sludges from the oil-agglomeration process scheme. Transportation costs are not included in the economic analysis, although a 20-day capacity for product storage is provided. The results of this analysis, therefore, will reflect on-site costs of agglomerated waste coal recovery.

A detailed breakdown of equipment and construction costs of the 50 TPH oil-agglomeration process scheme shown in Figure 12 is presented in Table 25. Many of the cost estimates in this table were obtained from EPA documents and chemical engineering literature [23,25-27]; all of the costs include installation except for pumps and external piping. All capital costs have been adjusted for inflation to July, 1980. Total equipment costs were estimated at 1.1 million dollars, more than half of which was expended for sedimentation basins ($319,800) and for the DAF unit ($255,900). Using the equipment cost total, the fixed capital costs for the oil-agglomeration scheme were calculated as shown in Table 26. The total fixed capital cost was estimated at around 3.7 million dollars.

Operating and maintenance costs are estimated in Table 27 for the 50 TPH coal agglomeration plant. These costs include operating labor, equipment

TABLE 25. EQUIPMENT AND UNIT PROCESS COSTS OF A 50 TPH OIL-AGGLOMERATION WASTE COAL RECOVERY FACILITY

Process	No. of Units	Unit Capacity	Unit Cost, $	Total Cost, $	Source Reference$^{(a)}$	Source Cost Index$^{(b)}$	Cost Index$^{(b)}$ July 1980	Cost, $ July 1980
I. Rapid Mix	1	3.0 MGD	3,800	3,800	EPA, 1972	136	269	7,520
II. Chemical Storage and Feed								
• Oil								
- Cone roof steel tanks	2	144,000 gal	22,000	44,000	Chem Eng, 1976	192	291	66,700
- Metering pumps	2	10 GPM	5,000	10,000	1979 Price Est.	274	328	12,000
• Soda Ash/Na_2S - Feed system	1	1.0 TPD	30,000	30,000	1980 Est.		269	30,000
III. Coal Agglomeration	2	3.0 MGD	14,000	28,000	EPA, 1972	136	290	59,700
IV. Static Flotation-Sedimentation	1	3.0 MGD	50,000	50,000	EPA, 1972	135	290	106,600
V. Resuspension Tank	1	1.5 MGD	2,500	2,500	EPA, 1972	136	290	5,300
VI. Dissolved Air Flotation Unit	1	1.5 MGD	120,000	120,000	EPA, 1972	136	290	255,900
VII. Dewatering Screen (130 ft^2)	1	30 TPH	36,000	36,000	EPA, 1972	136	290	76,800
VIII. Rapid Mix	1	4.1 MGD	4,600	4,600	EPA, 1972	136	290	9,800
IX. Alum Feeding Equipment	1	3.0 TPD	17,500	17,500	EPA, 1972	136	290	37,300
X. Flocculation	1	4.1 MGD	26,000	26,000	EPA, 1972	136	290	55,400
XI. Sedimentation Tank	1	4.1 MGD	100,000	100,000	EPA, 1972	136	290	213,200
XII. Coal Product Handling and Storage								
• Storage Area 1 (9700 ft^2)	1	30-day storage	97,000	97,000	1980 Est.	290	290	97,000
• Belt Conveyor 1 (200 ft long)	1	35 TPH	8,000	8,000	Chem Eng, 1967	109	290	21,300
• Front End Loaders	2		14,000	28,000	1980 Est.	290	290	28,000
XIII. Pumps								
• High Flow	2	4000 GPM	7,000	14,000	Chem Eng, 1976	221	328	20,800
• Sludge Handling	2	400 GPM	3,100	6,200	Chem Eng, 1976	221	328	9,200
• Low Flow	1	30 GPM	1,000	1,000	Chem Eng, 1976	221	328	1,480
					TOTAL EQUIPMENT COST			$1,114,000

(a) Reference Key: EPA, 1974 prices (23); Chem. Eng., 1976 prices (26); Chem. Eng., 1976 prices (27).

(b) Costs of unit processes and the coal product storage area include construction and installation labor. Installation costs of pumps and piping are not included. The CE Plant Cost Index was used to adjust all costs to 1980 prices (29).

TABLE 26. FIXED CAPITAL INVESTMENT OF A 50 TPH OIL-AGGLOMERATION WASTE COAL RECOVERY FACILITY

Item	Cost, \$[(a)]
Direct Costs	
Equipment and Installation	1,114,000
Instrumentation and Controls	144,800
Pumps and Piping Installation	345,300
Electrical (installed)	111,400
Service Facilities and Housing	612,700
Site Improvements	111,400
TOTAL DIRECT PLANT COST	\$2,409,600
Indirect Costs	
Engineering and Supervision	356,500
Construction Expenses (misc.)	378,800
TOTAL INDIRECT PLANT COST	\$735,300
TOTAL DIRECT & INDIRECT	\$3,144,900
Contractor's Fee	200,500
Contingency	401,000
TOTAL FIXED CAPITAL INVESTMENT	\$3,746,400

(a) July, 1980 dollars.

TABLE 27. ANNUAL OPERATING AND MAINTENANCE COST ESTIMATES FOR A 50 TPH OIL-AGGLOMERATION WASTE COAL RECOVERY FACILITY

			Annual Cost, $ (1980)
Labor:			
Operating Labor, 4 men/shift, $10/hr			$288,000
Supervisor, 20% of operating labor			57,600
Maintenance, 3% of direct plant cost (DPC)			72,300
		TOTAL LABOR	$417,900
Maintenance Supplies: 2% of DPC			48,200
Plant Overhead: 60% of total labor			250,700
Utilities: 150 kW electric power demand at $.03/kWh			32,400
Chemicals:			
	Dose	Chem. Cost	
Oil	0.024 ton/ton coal feed	$225/ton	1,944,000
Soda Ash/Na$_2$S	0.00083 ton/ton coal feed	$120.90/ton	36,100
Alum	0.0025 ton/ton coal feed	$102.30/ton	92,100
Property Tax: 1% of fixed capital investment (FCI)			374,600
		TOTAL	$3,196,000(a)

(a) O&M costs shown here do not reflect interest and depreciation on the FCI. These factors have been incorporated separately in the economics equations presented in the appendix.

repair and maintenance, chemicals, oil, and electricity. Electricity costs were based upon detailed electric power estimates for the process scheme as presented in Table 28. It is significant that the highest operating cost of the oil-agglomeration process scheme is in oil purchases amounting to almost 2 million dollars or 59 percent of the total annual operating cost.

A summary of assumptions and inputs used in this economic analysis is shown in Table 29. Equations used to calculate the cost of agglomerated coal recovery on a dollars per unit ton basis are given in the appendix.

The results of the economic analysis are presentd in Table 30. The total processing cost of the final oil-coal agglomerated product was calculated at \$19.42 per ton. Assuming the agglomerated coal product has an energy value of 14,000 Btu per pound, the per unit energy cost of product recovery is about \$0.69 per MM Btu (\$0.73 per GJ).

As shown in Table 30, the largest single cost incurred over the life of the coal separation system is the oil demand. About 58 percent of the per ton cost of coal recovery is traceable to the large purchases of oil that would be required for agglomeration. Thus, the economics of this system are highly sensitive to the dosages of oil used. Therefore, a reexamination of the trade-offs between oil dosage rates and product recoveries is needed to determine the profitability of the system at various levels of oil expenditures. Such an optimization, however, may result in reducing coal recovery efficiencies for the sake of controlling oil costs. A more detailed study would be needed to determine the potential for system improvements to reduce oil demand and to optimize the operation of existing coal agglomeration systems.

To further illustrate the relative importance of various parameters to the economics of oil-agglomeration coal recovery, a sensitivity analysis on each factor is presented in Table 31. A 25 percent increase in the cost of oil either through rising commodity prices or through increased dosages would increase the per unit coal recovery cost by more than 14 percent. In contrast, comparable increases in the costs of other chemicals, such as alum and soda ash, and in energy would result in less than a 1 percent change in oil-coal production cost. In extrapolating these data, it would appear that other chemical additions that may be required to improve process performance and control sulfide emissions would probably have a similar minimal effect on coal

TABLE 28. ELECTRIC POWER ESTIMATES FOR A 50 TPH OIL-AGGLOMERATION WASTE COAL RECOVERY FACILITY

	Equipment Item	Capacity	kW(a)
I.	Rapid Mix	3.0 MGD	6.8
II.	Chemical Feeding		
	Oil	10 GPM	
	Soda Ash/Na_2S	1.0 TPD	
III.	Oil Agglomeration	3.0 MGD	
IV.	Oil-Coal Separation Basin	3.0 MGD	
V.	Resuspension Tank	1.5 MGD	0.6
VI.	Dissolved Air Flotation	1.5 MGD	38.8
VII.	Dewatering Screens	30 TPH	0.1
VIII.	Rapid Mix	4.1 MGD	8.2
IX.	Alum Feed	3.0 TPD	
X.	Flocculation	4.1 MGD	
XI.	Sedimentation	4.1 MGD	
XII.	Pumps		29
XIII.	Miscellaneous, lighting, conveyors, monitoring equipment, etc.		67.5
		TOTAL	150

(a) Estimated from EPA literature (28).

TABLE 29. ASSUMPTIONS AND INPUTS USED IN THE ECONOMIC ANALYSIS OF A 50 TPH OIL-AGGLOMERATION WASTE COAL RECOVERY FACILITY

Parameter	Value	Comment
Annual Interest Rate[a]	14%	Highly subject to change
Annual Inflation Rate	12%	
Electricity Cost	3¢/kWh	Industrial user rate
Life of Facility	20 years	Estimated life of expendable materials
Life of Loan	20 years	Same as life of facility
Tax Bracket	48%	Federal income tax
Depreciation	Straight line	Taken over 20 years
Days of Operation	300 days/year	
Chemical Prices		1980 prices
Oil	$.90/gal	
Soda Ash	$120.90/ton	
Alum	$102.30/ton	

(a) In this analysis it will be assumed that the entire amount for the fixed capital investment will be borrowed.

TABLE 30. SUMMARY OF RESULTS OF THE ECONOMIC ANALYSIS OF A 50 TPH OIL-AGGLOMERATION WASTE COAL RECOVERY FACILITY

		Cost, $ (1980)
I.	Fixed Capital Investment	$3,746,400
II.	Total Payments on Loan (inflation adjusted)	4,473,800
III.	Lifetime Operating Costs	
	Labor and overhead	13,372,000
	Supplies	964,000
	Electricity	648,000
	Chemicals	
	Oil	38,880,000
	Soda Ash/Na_2S	722,000
	Alum	1,842,000
	Tax	7,492,000
	TOTAL OPERATING COST	$63,920,000
IV.	Lifetime Depreciation Credit (inflation adjusted)	$711,100
V.	Total Coal Recovery Over Lifetime = 3,484,800 tons	
VI.	Cost per Ton Product $\frac{(II + III - IV)}{(V)}$	$19.42

TABLE 31. SENSITIVITY OF THE COST OF OIL AGGLOMERATION COAL RECOVERY TO PERCENTAGE CHANGES IN SELECTED PARAMETERS

Parameter	Base Value(a)	% Change in Parameter	New Cost, $/ton Coal	% Change in Cost
Chemicals Cost				
Oil	$2.59/ton product(b)	+25	22.21	+14.4
Soda Ash/Na_2S	$.048/ton product	+25	19.47	+0.3
Alum	$.12/ton product	+25	19.55	+0.7
Capital Cost (Fixed)	$3.75 million	+25	19.69	+1.4
Operating Labor Overhead and Supplies	$1.09 million/yr	+25	20.99	+8.1
Energy Cost	$.19/ton product	+25	19.47	+0.3
Days of Operation/yr	300	-25	20.59	+6.0

(a) Base cost of recovered coal = $19.42/ton.

(b) "Product" is the resulting oil-coal mixture produced by the process.

recovery costs as long as total dosage levels remained modest (below 0.005 tons/ton coal input) and chemical costs averaged $200/ton or less. Thus, the economics of waste coal recovery are not greatly affected by the cost of chemicals other than the oil.

The sensitivity analysis also reveals that if capital cost estimates have to be adjusted upward by 25 percent, an increase of only 1.4 percent would be realized in the recovered coal product cost; a doubling of fixed capital cost would still mean a modest increase in product cost of less than 6 percent. These results indicate that the fixed capital costs should have only a minor impact upon the economics of oil-agglomeration coal recovery.

The second-most influential parameter controlling waste coal recovery costs was operating labor and associated overhead. A 25 percent increase in this parameter would likely predispose an 8.0 percent increase in product cost.

To summarize, this preliminary analysis would indicate several fundamental observations that generally characterize oil-agglomeration economics. The first observation is that the total fixed capital cost is far outstripped by the lifetime operating cost (labor + chemicals + electricity + oil + tax + supplies, etc.) by a ratio of more than 14:1. This rather large imbalance in the economics would suggest a great potential for cost improvement through further process research and development to reduce sensitive operating cost parameters, such as oil demand and labor requirements.

Second, a high amount of working capital would be needed to operate the oil-agglomeration system. For a 50 TPH plant, nearly 2 million dollars per year would be required for oil purchases alone. Of the $19.42/ton coal recovery cost in our 50 TPH example, about $18.34/ton (94 percent) would be attributable to operating costs other than the capital-related cost.

Finally, it should be recognized that the cost of $0.70 per MM Btu for the recovery of a high quality fuel will appear more and more attractive as world energy prices continue to soar faster than the rate of inflation, as has been the case over the past decade. In light of commercial fuel-oil prices now exceeding $6.50 per MM Btu in the U.S., a $0.70 per MM Btu cost for oil-agglomerated coal recovery from coal plant effluents may already represent an attractive economic opportunity for the coal industry.

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APPENDIX

ECONOMIC ANALYSIS EQUATIONS

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APPENDIX

ECONOMIC ANALYSIS EQUATIONS

I. Annual Capital Cost

$$AC = FCI \left[\frac{i}{1 - (1 + i)^{-N}} \right]$$

where: AC = Annual capital cost
i = Interest rate (decimal)
N = Life of loan, years.
FCI = Fixed capital investment

II. Total Capital Cost Over System Life

$$TCC = AC \left[\frac{1 - (1 + r)^{-N}}{\ln (1 + r)} \right]$$

where: TCC = Total capital cost, inflation-adjusted over N years, valued in 1980 dollars
r = Inflation rate (decimal).

III. Depreciation Over System Life

$$DC = TR \left[\frac{FCI}{N} \right] \left[\frac{1 - (1 + r)^{-N}}{\ln (1 + r)} \right]$$

where: DC = Total straight line depreciation credits taken over N years, inflation adjusted, valued in 1980 dollars
TR = Tax rate (Federal Income Tax).

IV. Total Operating and Maintenance Costs Over Life System

$$OC = N(M + L + E + C + T)$$

where: OC = Total operating and maintenance cost over N years, 1980 dollars
M = Annual maintenance labor and supplies
L = Annual labor cost plus overhead
E = Annual electricity costs
C = Annual chemical costs
T = Annual Property tax.

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V. Cost of Oil-Agglomerated Coal Recovery

$$CE = \frac{TCC + OC - DC}{N \times EP}$$

where: CE = Cost of coal recovery in 1980 dollars per ton oil-coal product
EP = Annual output of oil-coal product, tons/year.